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A STUDY OF THE PROCESS OF SECURING ROSIN FROM WOOD BY EXTRACTION WITH A PETROLEUM SOLVENT

BY

HAROLD R BOEHMER

A THESIS SUBMITTED FOR
THE DEGREE OF BACHELOR OF SCIENCE
CHEMICAL ENGINEERING COURSE

University of Wisconsin

1915

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A STUDY OF THE PROCESS OF SECURING ROSIN FROM WOOD BY EXTRACTION WITH A PETROLEUM SOLVENT.

INTRODUCTION.

Resinous Wood Utilization.

The problem of obtaining as much valuable material and as little waste as possible from our forests is becoming of greater importance each year, and as a result, every means that will tend to increase the value of the products and thereby reduce the actual wood waste is being tried. It is stated, that of the total wood standing, only 15 to 20 percent reaches the market as lumber. The other 80 to 85 percent consists of small trees, limbs, edgings, etc. In many plants this waste product is burned so as to get it out of the way. Of course, some of it can be used as fuel to furnish power for the mill but this will consume only about 40 to 45 percent of the total waste.

In resinous wood industries there are two scources of material for derived products. First, there are the small trees, and mill waste which constitute the live or freshly cut wood. Then there is the dead wood in the forest which is known as lightwood due to the fact that it burns with a luminous flame. This is the wood which has been dead for ten years or more, and from which all but the resinous material has decayed. The stumps can be classed as light wood also. It is not uncommon to have

lightwood which has a resin content of 40 percent.

Several processes are now in operation for the utilization of these resinous wood wastes. Destructive distillation is carried on in some localities. In this process, the wood is heated in retorts out of contact with the air and all the volatile matter passed through condensers. The products of this process are a non-condensable gas, light oils which contain wood turpentine and pine oil, pyroligneous acid, wood tar, and a residue in the retort of charcoal. The gas can be used as a fuel but is allowed to go to waste in some cases. The turpentine and pine oil, when purified find a ready market. The pyroligneous acid is too weak to work up and is sold as a disinfectant. The tar is refined so as to give oils which, when combined with the heavy distillates from the light oil fractions, will give wood creosote and disinfectants. In this process the rosin is decomposed and the light oils contain the products of decomposition known as the rosin oils. These are also commercially important. The charcoal is of good quality and can be used in the place of hard wood charcoal.

The turpentine obtained by the destructive distillation process cannot be purified from the peculiar odor due to the decomposition products of the wood. In order to overcome this, the process of steam distillation was introduced. The richest lightwood and stumps are used along with the mill waste. After being cut into chips, the wood is put in suitable retorts and steamed. The mixed vapors of steam and turpentine pass off to

condensers. The products in this case were turpentine and the residual wood still containing rosin.

The " bath " process accomplishes the same end by subjecting cord wood to a treatment with a hot bath of a non-volatile pitch or rosin. The bath was kept at a temperature near the boiling point of the oils and after being extracted, the volatile material was distilled from the solvent. It is also claimed that some rosin is obtained by this process.

The alkali process consists of removing the rosin from the wood as the sodium salt of the resin acids. By treating the wood with sodium hydrate or sodium carbonate, these salts are formed and are soluble in hot alkali solutions. After the solution is removed from the wood, it is allowed to cool and the resin soaps precipitate out. This sodium resinate has a possible use in the manufacture of soap. Treatment with acids will form the rosin again. The resinates can be distilled destructively to obtain the rosin oils.

A modification of the alkali process has been worked out by Whitaker and Bates of the Chemical Engineering Department of Columbia University.(a) They steam the wood in the presence of dilute alkali to obtain the turpentine oils. It is then cooked in stronger alkali to saponify the resin. This soap solution is drawn off and the rosin salted out by concentrating the solution with alkali. The wood residue is then digested with this con-

---

(a). Chemical Utilization of Southern Pine Waste.-M.C.Whitaker and J.S.Bates. Jour. Ind. and Eng. Chem. April 1914.

centrated alkali solution and converted into pulp.

Within the last ten years the solvent process has been developed to such an extent that it is in operation in several localities at the present time. In this process the wood is first steamed for the recovery of the volatile oils and is then subjected to the action of a volatile solvent for the removal of the rosin. After extraction, the wood is steamed again for the recovery of all the solvent. The solution of rosin is then distilled and the solvent reclaimed while the rosin is ready for the market. If this method could be operated satisfactorily, it would be possible to use the lightwood - which is too resinous for lumber,- and to obtain from it rosin,turpentine,pine oil, and the residue of extracted wood which might be utilized in some other process.

The first reference to experiments on this method is given by George Walker who claims to have built a plant at Conway, South Carolina in 1905.(a) His process was not patented until 1908.(b) However Frankforter of the University of Minnesota applied for a patent in 1906.(c) No details of the Walker process were given. He steamed for the volatile oils and then recovered the rosin with a volatile solvent, with the principle based " on the principle of the well known Soxhlet extractor."

(a) Methods of Utilizing Wood Waste.- Geo. Walker.

Chemical Engineer 15 51.

(b) Yaryan U.S.Patent No. 922369

(c) Frankforter U.S.Patent No. 918990

In 1912 this plant was producing 200 barrels of rosin per week and a considerable supply of turpentine.

According to Teeple, there were eight plants in operation in 1913 which were using this system.. These plants were located in the states of Mississippi, Alabama, Florida, South Carolina, and Michigan.(a)

Purpose of Work.

Only a few of these plants are operating successfully. The greatest trouble seems to be in the large solvent loss which according to Teeple amounts to 20 to 25 gallons per cord of wood of 4000 pounds. The plants also obtained low efficiency of extraction. It seems that few attempts have been made to dry the wood after steaming and before exaraction. As the moisture in the wood seemed to hinder the extraction, steaming before extraction was thought objectionable. The above authority concludes, that due to the decreasing price of the products, the increasing price of the solvent,the loss of solvent, and also the low efficiency of extraction, some changes in the nature of the process are necessary in order that it may remain commercially successful. For instance,it might be modified so that the residual wood after extraction could be utilized for another process such as pulp manufacture.

It was for this reason that a study of the process and the effect of varying the conditions under which it is carried out

---

(a) Utilizing Wood Waste.- J.C.Teeple
Jour. Ind. and Eng. Chem. 1913

was begun. Two things were sought. First, it was desired to determine those conditions under which the highest yield of rosin and turpentine could be obtained. Second, it was desired to locate the loss of solvent and to try to prevent it.

SCOPE OF STUDY.

The wood used in these experiments was southern long leaf yellow pine stumps. These compared very favorably in most cases with lightwood as they had been cut for some time.

The variables studied were as follows:-

1. The effect of time on extraction.
2. The effect of the pressure of extraction.
3. The effect of the oleoresin content of the wood.
4. The effect of omitting the vacuum for drying the steamed wood.
5. The effect of the size of the chips.

It was also possible to obtain the combined effect of two or more of these variables.

The first ten runs were made on wood that had been cut into 3/16 inch chips in a pulp chipping machine. The last two runs were made on 1/2 to 5/8 inch chips. All runs included steaming for the removal of the turpentine and, in all cases excepting run 10, a vacuum was drawn after that to dry the wood. Run 10 was to determine the effect of moisture in the wood and hence the vacuum was omitted. The details of the experiments are as follows:-

On 3/16 inch chips.

Run 1 15 minutes extraction, 0 pounds pressure.
Run 2 45 minutes extraction, 0 pounds pressure.
Run 3 15 minutes extraction, 30 pounds pressure.

Run 4 45 minutes extraction, 30 pounds pressure.
Run 5 15 minutes extraction, 55 pounds pressure.
Run 6 15 minutes extraction, 30 pounds pressure.
Run 7 15 minutes extraction, 0 pounds pressure.
Run 9 15 minutes extraction, 30 pounds pressure.
Run 10 15 minutes extraction, 30 pounds pressure.

On 1/2 to 5/8 inch chips.

Run 11 15 minutes extraction, 30 pounds pressure.
Run 12 45 minutes extraction, 30 pounds pressure.

Due to several accidents, the results of run 8 could not be determined and hence it had to be repeated in run 9.

Runs 1,2, and 3 were on wood of about the same oleoresin content. Runs 4, 5, and 6 were on wood of about the same oleoresin content but the wood was much richer than the first. The remainder of the tests were on wood of similiar resin content but which was lower than the other wood used.

CONDUCT OF TESTS.

The requirements of the extraction were that it should be carried out in a continuous extraction battery. Each unit was to receive exactly the same treatment. In all cases, the most nearly saturated solvent was to be run onto fresh chips and the freshest solvent was to be run onto chips that were nearly extracted. This was brought about by a system of three retorts and six solvents which were used in a test as shown in the table below

Table 1.

PRELIMINARY			RUN 1			RUN 2
RETORT 1	RETORT 2	RETORT 3	RETORT 1	RETORT 2	RETORT 3	RETORT 1
Charged	Charged	Charged				
Solv I						
Solv II	Solv II					
Solv 111	Solv III	Solv III	Charged	Charged	Charged	
Solv IV	Solv IV	Solv IV	Solv IV			
	Solv V	Solv V	Solv V	Solv V		
		Solv VI	Solv VI	Solv VI	Solv VI	Charged
			Solv VII	Solv VII	Solv VII	SolvVII
				Sol VIII	Sol VIII	Sol VIII
					Solv IX	Solv IX
						Solv X

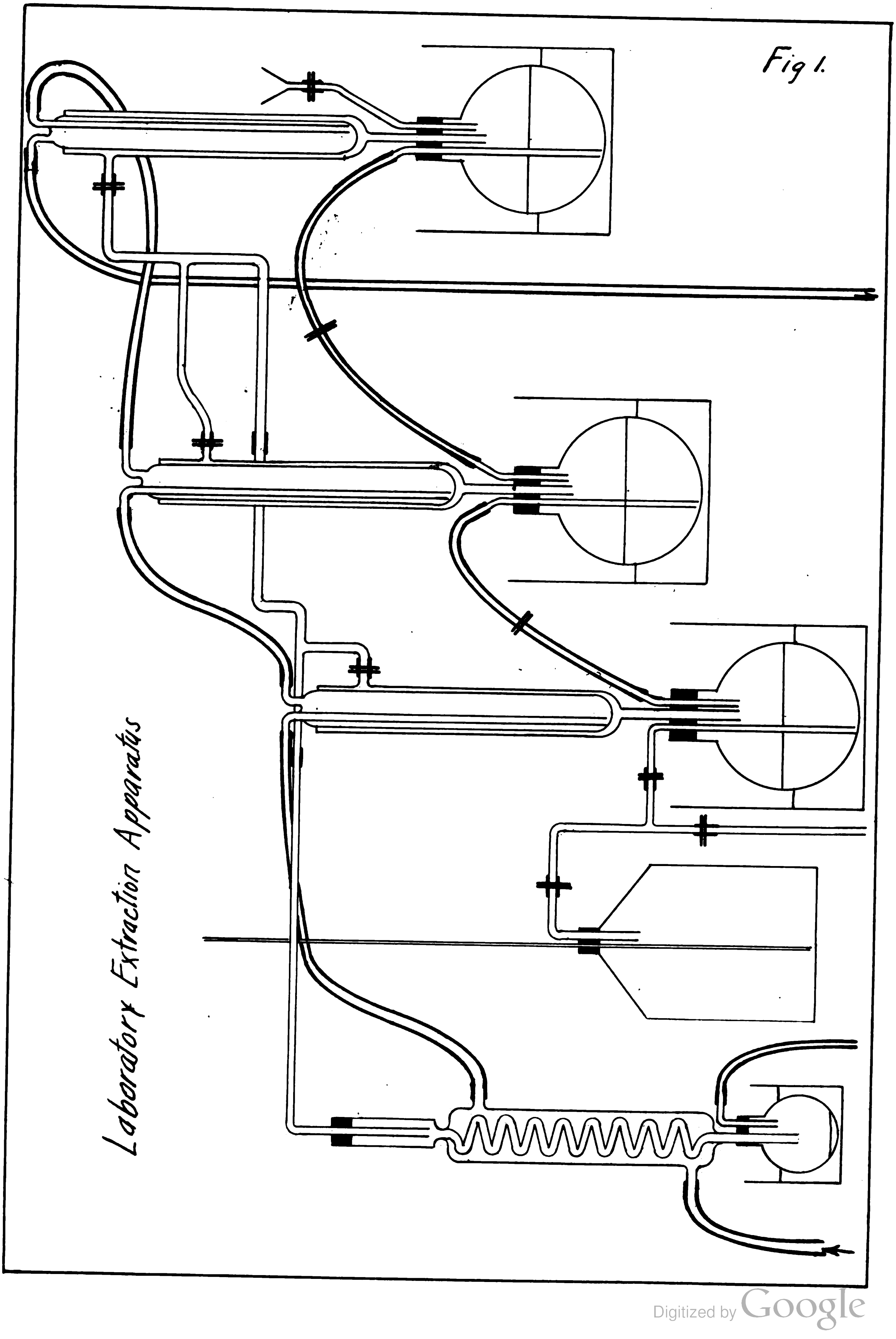
Table Showing Method of Extraction.

It is seen by the above table that each charge has four washes and that in each test, three solvents must be refined before they are used again. Three solvents are carried over from one run to the next. This is the nearest approximation to the commercial continuous proceedure that could be obtained.

Laboratory Tests.

In order to determine, if possible, what might be the results to be expected on the semi-commercial apparatus, it was decided to carry out a few extractions on a small scale in such an apparatus as could be built out of laboratory glassware. An apparatus was designed which would be comparable with the larger apparatus to be used later and is shown diagrammatically in Fig.1. The flasks used were of 1000 c.c. capacity. The tubing was all about 0.75 cm. internal diameter. All stoppers were of cork, free from unsound places, and after the apparatus was set up, all joints were covered with collodion. At first, it was thought unnecessary to include the coil condenser, but the results show that it had some effect in the condensation of the more volatile solvent. Four tests were run on this apparatus but it was found necessary to combine the first two in order to calculate the results. The charge of wood was about 100 grams of 3/16 inch chips in each flask. Extraction was carried out as shown in the above table. No preliminary steaming was carried out for the recovery of turpentine but steaming was done for the recovery of solvent after extraction. The baths, in which the flasks were heated, were concentrated solutions of calcium

Fig 1.



Laboratory Extraction Apparatus

chloride. The flask under the coil was kept in an ice pack.

Results of Laboratory Tests.

Table 2.

Run Number.	1&2	3	4
Time of extraction	10 min.	20 min.	10 min.
Efficiency	77.9%		75.5%
Solvent retained	18.9%	14.8%	14.2%
Solvent caught by coil	1.8%	0.4%	0.7%
Solvent loss	10.40%	3.90%	0.93%

The results of the laboratory extractions as given in the above table show quite conclusively that it is purely mechanical difficulties which hinder the extraction process. It is evident that the solvent loss can be reduced to a very low value by the proper precautions. The coil condenser shows that a portion of this solvent vaporizes readily and is not condensed by ordinary methods. The amount of solvent retained in the chips after they had been drained is considerably greater than in the tests on the semi-commercial apparatus but this may be due to the inefficient method of removing these solvents by siphon.

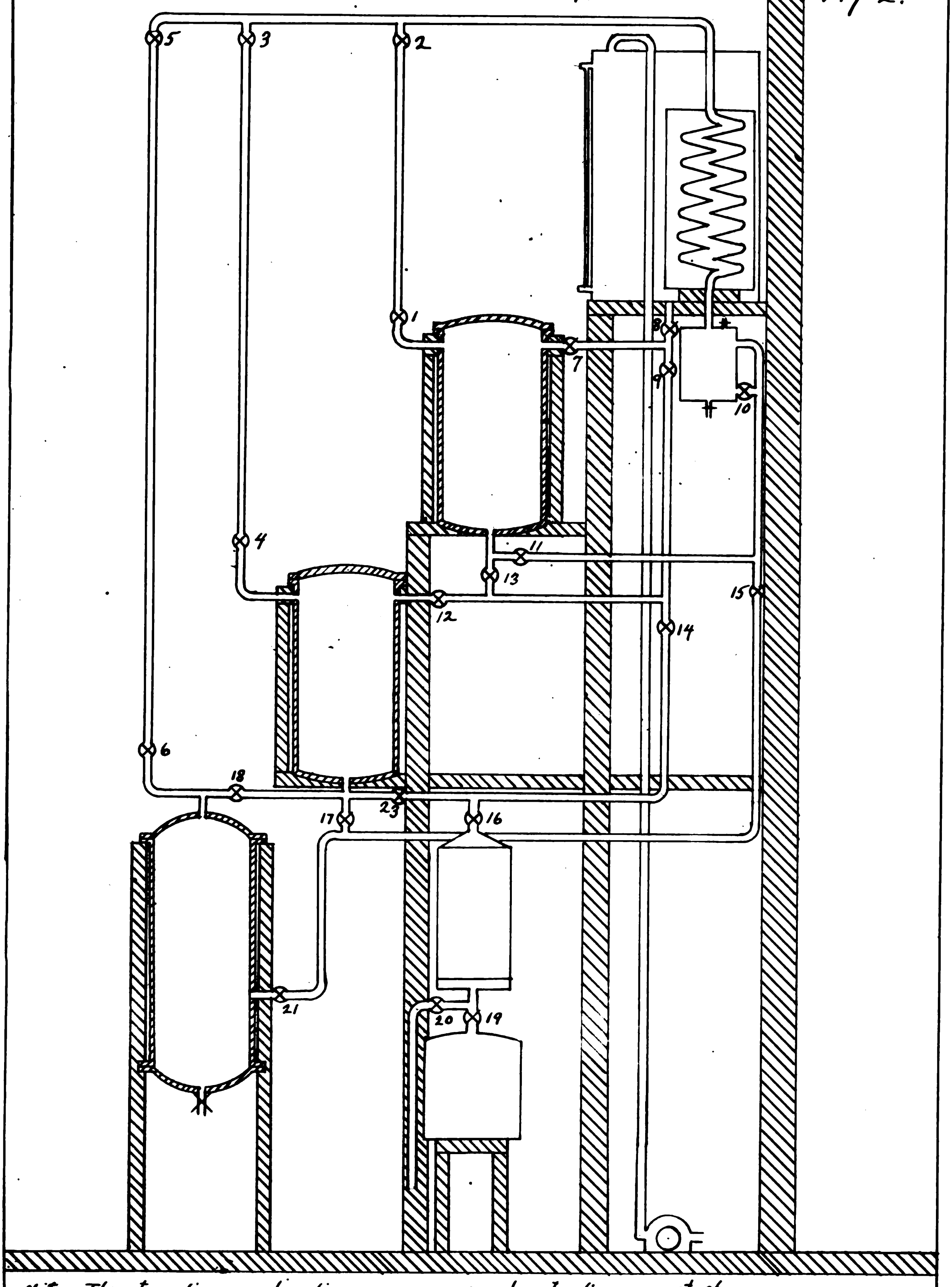
Semi-Commercial Tests.

Apparatus. The laboratory extractions were made only as a means of preliminary study of the problem. They were expected to show up any difficulties in the larger apparatus and to show the possible yields and solvent losses.

The method used in the semi-commercial tests was that known in chemical industries as the diffusion battery system. The

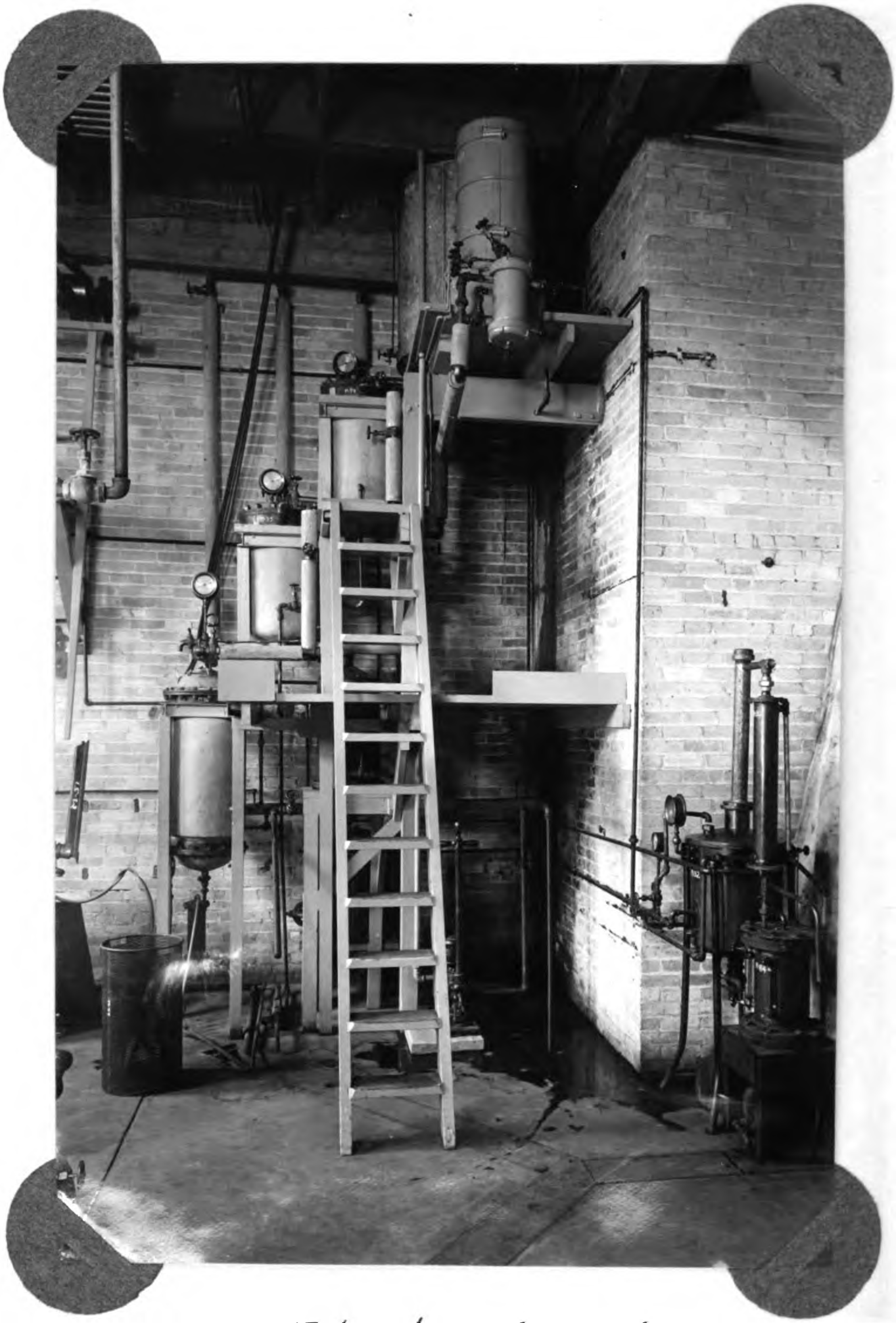
Semi-commercial Extraction Apparatus

Fig 2.



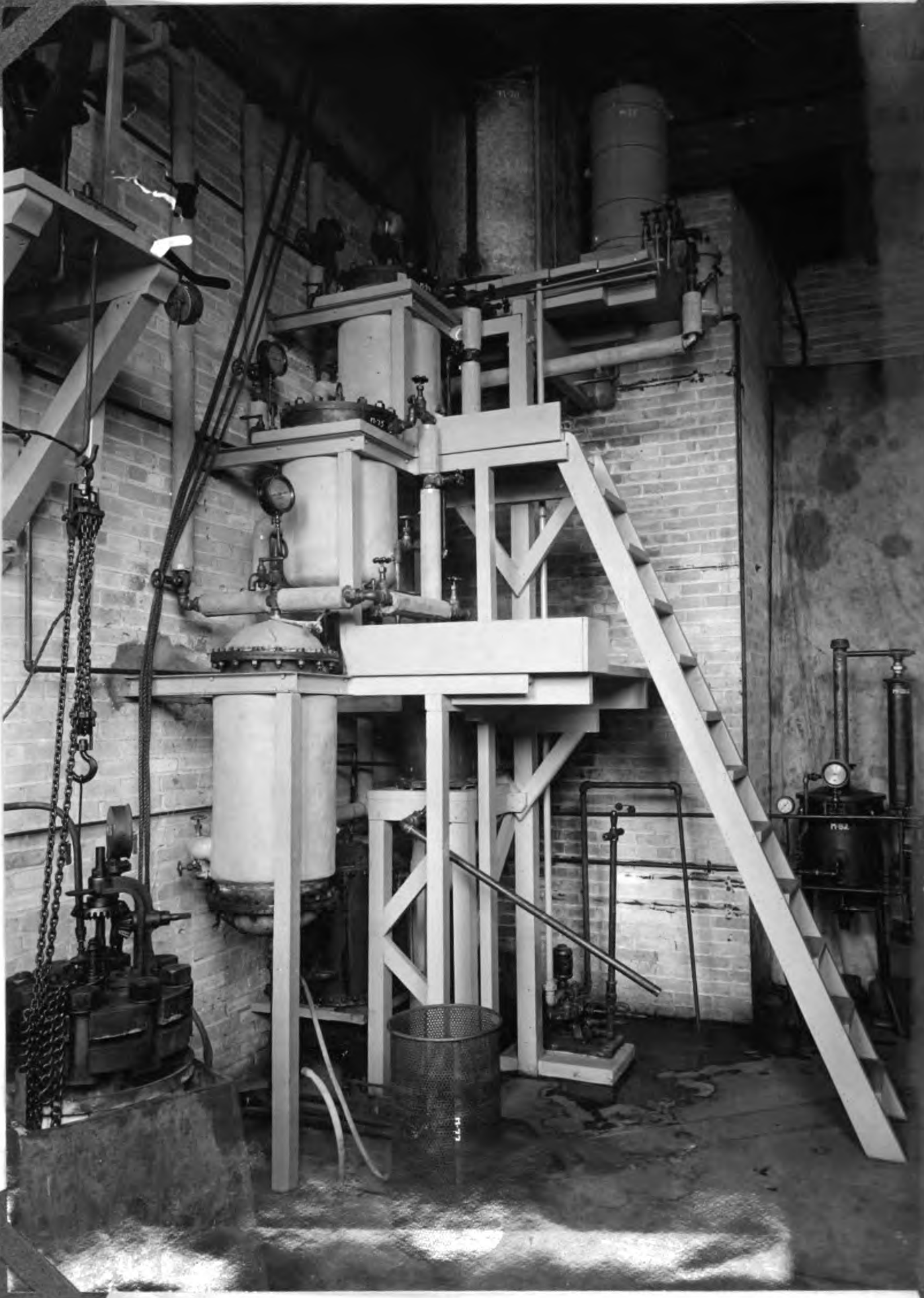
Note:- The steam lines, equalizer lines, pressure gages, and water lines are not shown.

Fig 3.



Extraction Apparatus.

Fig 4



Extraction Apparatus

apparatus used was designed and built at the Forest Products Laboratory for experimental work along this line. It is shown diagrammatically in Fig.2, and also in the photographs in Fig.3 and Fig. 4. The diagram does not show the actual location of all the pipe lines, but was made so as to show the connections with the various retorts and condensers. The solvent flowed by gravity after it had once been elevated to the storage tank.

The storage tank was of galvanized iron and had a capacity of 150 gallons. It was set on a platform 13 feet, 4 inches from the floor and had two openings. One of these was in the top and was connected by a pipe with the force pump on the floor for elevating the solvent. The other opening was in the bottom and was used to carry the solvent to the retort in which it was desired. In front of this, and on the same platform, was a condenser of the coil type. The vapor coil was of copper. The cooling water was in an open wooden tank so that the coil could be examined for leaks readily. Just below the condenser was a trap designed for the separation of any water from the solvent when the mixture was condensed. The condenser and trap corresponded to the reflux condensers of the laboratory apparatus in effect. The first retort was suspended from this platform also. It was wrought iron and was 36 inches long by 16 3/4 inches in internal diameter. This retort, like the other two was fitted with a steam coil for heating the charge and also a single loop of perforated pipe for steaming the charge. There were three openings in the retort. Two of them were near the top and

opposite each other. One led upward and over to the coil condenser and was used to carry the vapors off when the charge was being boiled. The other opening led through a pipe to the bottom of the storage tank and also to the top of the tubular condenser. A third hole in the bottom led through a pipe to the line from the trap. This was for returning the condensed vapors to the retort and closing the refluxing system.

The second retort was set on a platform 10 feet from the floor and was of the same dimensions as the first. It was connected to the system the same as the first retort. The third retort was of copper and was 42 inches long by 14 inches inside diameter. An opening in the middle of the top, by means of a tee.,connected with the line to the coil condenser and also the line from the storage. . An opening in the side near the bottom of the retort led to the trap. As it was impossible to drain the solvent from the third retort through the big tubular condenser, a smaller condenser was attached to the bottom of the retort so that the drain was only 18 inches from the floor. Under the second retort was a large tubular condenser which was connected to the bottoms of the two upper retorts and the tops of all three. The pipe from the bottom of this condenser led through a tee to a drain and also to the receiver for vacuum distillation. The receiver was a 35 gallon steel tank connected to a vacuum pump. The solvent was kept in 35 gallon cans fitted with handles and encased in wooden frames so as to prevent injury. The covers screwed on fairly tight and there was an

ordinary faucet for draining off the solvent.

Each retort was fitted with a basket made of sheet iron punched with 1/2 inch holes. The baskets in the two upper retorts were 30 inches long by 15 1/4 inches in diameter while the one in the bottom retort was 34 inches long by 13 inches diameter. A block and tackle was suspended frm the ceiling to lift off the covers and lift out the baskets. Each retort was fitted with a compound pressure and vacuum gage. This was not attached directly to the retort but to the vapor line just outside the retort. It was found desirable later to include a set of pipes to equalize the pressure between the retorts and the trap. By connecting each retort in this manner- namely by a small pipe leading from the outlet of the retort to the top of the trap-, the pressure on the condensed vapors was always equal to that in the retort.

Vacuum distillations were carried out by use of a small vacuum pump operated by an electric motor. It was a wet pump and was capable of drawing a vacuum of 20 inches of mercury on the extraction battery.

The actual proceedure followed in an extraction was as follows. The wood was first chipped in a pulp chipping machine and after being sampled, was put into the baskets. These were filled about two thirds full. After the baskets were lifted into place and the covers bolted down, the apparatus was ready. In the following explanations, it is assumed that all other valves are closed excepting those designated.

The first step in the process was the removal of the volatile oils by steam distillation. Previous investigation has shown that the yield of turpentine in increased with increased pressure of distillation, and so pressure distillations were employed.[a] After the retorts had been closed, steam was turned on in the heating coil and also in the open coil of retort 1 so that the pressure would rise slowly. When it had reached the required amount, valves 9, 14,16, and 20 were opened wide and valve 7 was varied so that the condensate would come cool. Then the steam in the open coil was varied so that the pressure would be that desired. This distillation was carried on until the distillate was practically all water. This required at least an hour. Occassionally, the retort was "blown off". This consisted of opening valve 7 for an instant and then closing it until the pressure had risen to the normal value. Such a proceedure seemed to dislodge pockets of distillate that might collect in the pipe system. When the distillation was finished, the steam was turned off and the pressure was allowed to fall slowly. Retort 2 was then steamed in a similiar manner, the vapors passing off through valves 12, 14, 16, and 20. Retort 3 was then steamed and the vapors conducted through valves 18, 16, and 20. The chips were now saturated with water and, in order to remove this, it was necessary to draw a vacuum. In some cases the retorts were dried separately and in other cases

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(a) Distillation of Resinous Wood by Saturated Steam.
L.F.Hawley and R.C.Palmer Forest Service Bulletin 109.

they were all dried at the same time[a] it did not seem to make any difference for the vacuum pump would draw a vacuum of 29 inches in either case. The valves opened for vacuum distillation for retort 1 were 13, 14, 16, 19, and 22. For retort 2 they were valves 17, 16, 19, and 22 and for retort 3 the valves open were 18, 16, 19, and 22. The vacuum was drawn for 90 to 150 minutes per retort or about 4 hours for the entire system.

The wood was now ready for extraction for the removal of the rosin. Each solvent was weighed and pumped up to the storage tank just before it was to be used. The empty can was then weighed and the amount of solvent used determined by difference. Considering each solvent in the storage tank at the time when it is to be used, the procedure will be described. Solvent I, which had been through three retorts in the previous run, was run into retort 1 by opening valves 7 and 8. Then valves 1,2, and 24 were opened so as to allow the air to escape to the trap. It was allowed to escape through a small pet cock. When it was thought that all the solvent has been transferred to the retort, valve 7 was closed and valves 9, 14, 16, and 20 were opened. If practically no solvent was drained off, it was decided that it had all been transferred to the retort. Valve 8 was then closed and the steam turned on in the heating coil. The boiling solvent would then force vapors up through the vapor line designated by valves 1, 2, and 24. These would be condensed and flow into the trap. The extraction was said to have begun when the gauge in the trap showed a rising liquid level. The pet cock in

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(a) Steam was turned on in the closed coil during vacuum drying.

the trap was left open until boiling had begun. It was then closed and the equalizer line opened also. Then the refluxing line through valves 10 and 11 was opened. The boiling should then proceed without any appreciable change in the height of the liquid level in the gauge in the trap as the solvent was flowing back to the retort as fast as it was being condensed above the trap. After the extraction was complete, the solvent was drained off through valves 13, 14, 16, and 20. The pet cock in the trap was opened to allow the vacuum formed by the solvent to be broken. Solvent II, which had been through two retorts in the previous run was then run into retort 1 as before and the extraction carried out. When the time for boiling was completed, this solvent was transferred to retort 2 by first opening valve 12 and 13 and later valves 4 and 3 for the escape of air. The transfer was made in some cases equally well by way of valves 13, 15, and 23 with valves 12, 14, 16, and 20 for the escape of the air. As before, when it was though that the solvent had all been transferred, the valves were opened through the drain as a test. If an unusually long time was required, it was evidence that some of the valves were clogged and should be cleaned. Valves 1, 2, 10, 11, and 13 were then closed and also the equalizer to retort 1. Retort 2 was then brought to boiling and the refluxing line through valves 10 and 15 was opened. After extraction, this solvent was drawn off through valves 17, 16, and 20.

Solvent III which had been through one retort in the prev-

ious run was then run into retort 1 and the extraction carried out. It was transferred to retort 2 and the extraction carried out there also. It was then transferred to retort 3 by way of valves 17 and 18 or 23 and 21 with the air escaping through valves 6 and 5 in the first case or through valves 18, 16, and 20 in the second case. The boiling was then carried out as before with the refluxing line through valves 10, 15, and 21, After extraction, the solvent was drawn off through the valve in the bottom of the retort. The next three solvents were fresh solvent free from rosin. Solvent IV was allowed to extract all three retorts in succession and when drained was called solvent I of the next run. Retort 1 had now had four washings and was not treated further. Solvent V was run into retort 2 through valves 8, 9, 13, 12, and retorts 2 and 3 were successively extracted. This solvent when removed was called solvent II of the next run. As retort 2 had now had four washings, it was not further treated. Solvent VI was then run into retort 3 by way of valves 8, 9, 14, and 18. After extraction, this solvent was drained and called solvent III of the next run. The extraction had now been carried out as cutlined in the table on page 10 and in such order that the richest wood was treated with the most saturated solvent and the poorest wood was treated with the freshest solvent.

Some deviation from this proceedure was necessary when extraction was carried out under pressure. The method will be described for retort 1 only but it was similiar for all three.

After the solvent had been run in, the heating coil was turned on and the solvent was allowed to come up to boiling as in the atmospheric extractions. When the liquid column in the trap began to rise, thus showing that boiling had begun, valve 1 was closed tightly until the pressure in the retort was as high as desired. Then that valve was opened slightly. The equalizer line was opened slowly and then the refluxing line was opened. Then after allowing a minute for conditions to come to equilibrium, valve 1 was controlled until the pressure in the retort was that desired. Just before the equalizer line is opened, the pressure in the retort will fall but it usually rises to the original value in a short time. Extreme care must be taken when pressure extractions are being made that all the air is out of the system before the pet cock in the trap is closed. It was quite easy to maintain a pressure of 30 pounds but it was very difficult to keep the pressure at 55 pounds. It is probable that the steam pressure in the heating coil was hardly high enough to keep up this pressure. Also the radiation from the vapor lines was large.

The next step in the process was the recovery of the solvent that was retained in the chips. This was done by the same method of steam distillation as was used for the recovery of the turpentine oils. After the solvent had been recovered, a vacuum was drawn on the system to remove some of the water but mainly so as to determine if all the solvent had been removed. The treatment of the wood was then completed and the retorts

were opened and the baskets lifted out and weighed.

Solvents I, II, and III were then distilled for the recovery of the rosin dissolved in them and also for the purification of the solvent for further use. This was carried in a copper still which had a capacity of 15 gallons. The still is shown in the photographs of the extraction battery. The dephlegmator was replaced by a simple still head made of three inch pipe. This was connected to a tubular condenser which had two outlets; one to a drain, and the other to a receiver for vacuum distillation. The pot of the still was fitted with a closed steam coil for heating and an open coil for direct steaming. It was found advisable to use not more than thirteen gallons of solvent in the retort at a charge as there was considerable foaming. Starting the distillation required considerable care, especially when the solvent was very dilute. The steam pressure had to be below one pounds gage pressure in order to prevent the solvent boiling over. As soon as the steam pressure in the coil was 45 pounds, the distillation was stopped and the still recharged. A higher temperature than that corresponding to this steam pressure was not desired. After all the solvent had been removed that would distill below this temperature (145 deg. C.), the direct distillation was stopped and steam distillation carried out until the distillate was practically pure water. A vacuum of 26 inches was then drawn on the rosin to free it from all the moisture and the temperature was kept at about 150 deg.C. While still hot, the rosin was drained from the bottom of the still

pot and weighed. However these weights could not be used in the results of the experiments as considerable rosin adhered to the walls of the still.

Methods of Analysis.

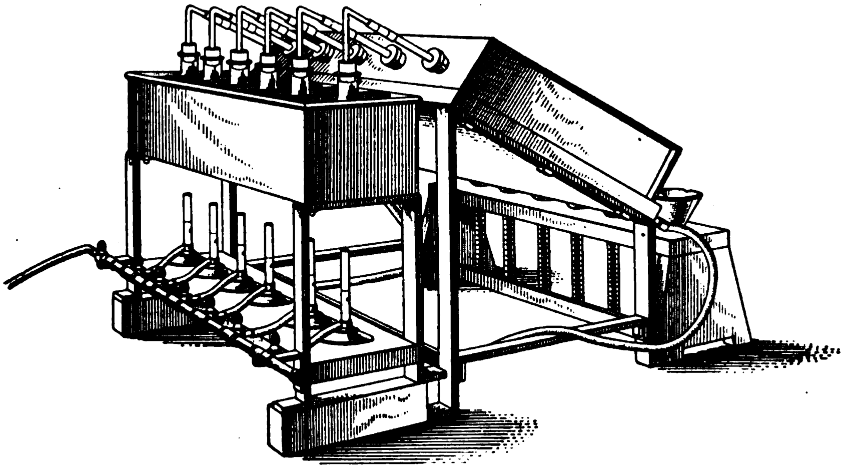
The samples of wood for analysis were taken by the quarter method used extensively for materials which are liable to vary in composition throughout the mass. The final sample was about 100 grams and was kept in ground glass stoppered bottles. The solvents, after removal from the system, were thoroughly stirred and samples of about 300 cubic centimeter were taken and stored in glass stoppered bottles. In order to eliminate the errors due to the evaporation of moisture from the wood and its condensation on the walls of the bottles, the samples for analysis were taken as soon after sampling as possible and they were all weighed out at the same time.

ANALYSIS OF WOOD.

Moisture in Wood. The determination of moisture in wood at first seems to present a difficult problem, Resinous wood, such as was used in this work, will contain water, turpentine, and pine oil besides the woody material. All of these are volatile excepting rosin which can be considered practically nonvolatile at these temperatures. Hence drying the wood at 110 deg. C. would not be an accurate method of determining the moisture as some of the other volatile matter would be removed. However, it would not be possible to remove all the volatile matter by this method. The method used in this case was that described for the determination of moisture in wood preserved with volatile

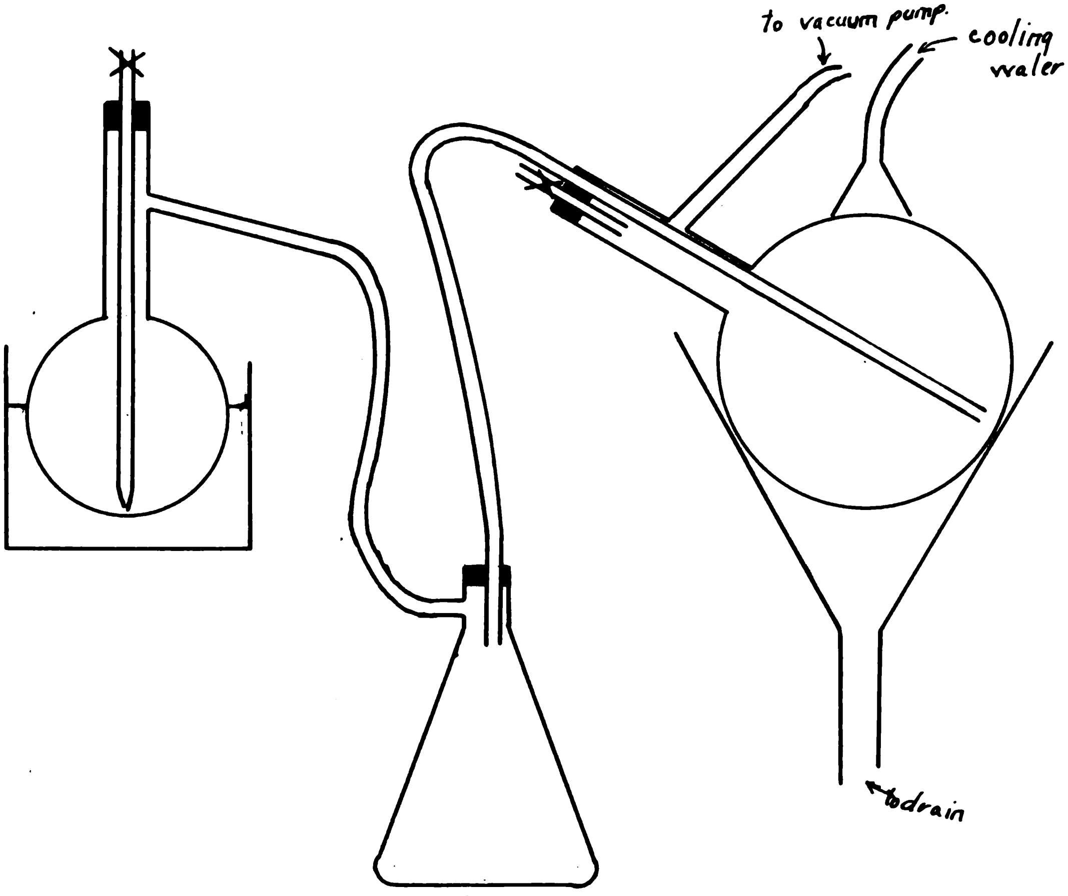
Fig 5

Appatus for Moisture Determination



Distillation Apparatus

Fig 6.



compounds.[(a)] It depends on the fact that when water and an oil which is immiscible with it are boiled, they will distill at a temperature below the boiling point of the lower. When the mixture has been distilled, the turpentine and other oils will dissolve in the oil and the water will float on the top. It can be measured and thus the moisture content determined. In this case water saturated xylol was used. It was prepared by taking pure xylol and heating and shaking it with water. The xolkl was then separated from the water and was ready for use. The apparatus used is shown in Fig. 5 . The flasks are immersed in an oil bath and the vapors are carried to condenser tubes encased in a copper water jacket. The distillate was caught in specially graduated cylinders reading to tenths of a cubic centimeter.

If the wood seemed quite dry, it was ground to the fineness of sawdust and then a sample of about 15 grams was weighed out in a tight weighing bottle. It was transferred to the flask and covered at once with xylol. The distillation was carried out slowly until the distillate came clear. The water on the side of the cylinders was brushed off with a "policeman" and the amount of water read off at once. When the wood was quite wet or when the chips were large, grinding would allow a part of the moisture to evaporate. In these cases, the wood was weighed out as received and covered with xylol. After standing for not less than 24 hours, the distillation was carried out as before.

(a) The Estimation of Moisture in Creosoted Wood- A.L.Dean

Forest Service Circular 134.

On the 5/8 inch chips this was found to remove all the moisture in one distillation.[a]

Rosin. A control extraction was carried out in the well known Soxhlet apparatus. An average sample of the material prepared for the moisture determinations was ground and put into a weighed alundum thimble. After weighing, the thimble was placed in the extractor and the wood was extracted with chloroform for about ten hours. When the extraction was complete, the thimble was drained, dried at 110 deg. C. and weighed. The loss in weight was then moisture, rosin, and volatile oils. The extractor was rinsed with pure chloroform and the combined liquid transferred to a 250 c.c. distilling flask fitted for vacuum distillation. The apparatus used is shown in Fig 6 and will be described later. The bath was kept at 150 deg. C. and the chloroform was distilled in a vacuum. When the rosin was apparently dry, steam was allowed to pass through it under slightly reduced pressure for the removal of the high boiling volatile oils. Steam distillation in a partial vacuum gave a violent agitation of the rosin and insured all the oil being removed. The rosin was then dried in a vacuum at 150 deg. C. to constant weight. As it was impossible to obtain constant weight even after six dryings of half an hour each, it was decided to dry until the loss in weight between any two consecutive weighings was less than 8 milligrams.[b] The moisture in this sample of wood was determined

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(a) In run 11, a second distillation was made over xylol after the chips had stood for 2 1/2 days and no water was obtained.

(b) Continued heating probably caused decomposition of rosin.

from the previous moisture determination. As the rosin and moisture was known, it was possible to determine the amount of volatile oils by difference.

Solvent Analysis. The determination of the amount of rosin in the solvent was carried by a simple vacuum distillation. The apparatus used is shown in Fig. 6 . The filter flask was used as a trap in case the solvent should boil over but also acted as a condenser to some extent. The vacuum could be broken instantly by opening a pinch cock in the stopper of the condenser proper. A sample of 200 grams was weighed out into a weighed half liter distilling flask and the apparatus was set up at shown. The vacuum was drawn carefully at first but after boiling had once begun, it would proceed with no trouble. The oil bath was kept at 150 deg. C. After the vacuum had been drawn for some time so as to insure that the rosin was dry, the flask was weighed. The increase in weight was then rosin. It was possible by this method to boil off the solvent which was about 225 c.c., in 20 minutes and to dry the rosin in 15 minutes more.

Crude Turpentine Analysis. The analysis of the crude turpentine was made according to the method used by the Forest Service. (a) It consists of distilling a sample of about 500 grams through a Hempel column and taking fractions whose boiling points and specific gravities are determined. Wood turpentine usually contains the following constituents:-

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(a) Wood Turpentines- Their Analysis, Refining, and Composition. L.F.Hawley. - Forest Service Bulletin 105.

	Pinene.	Dipentene.	Pine oil.
Boiling Point, deg.C.	155	175	205-210
Specific Gravity	0.858	0.849	0.945

From this table, it is seen that the boiling point of dipentene is higher than that of pinene and it specific gravity is lower. Hence if these compounds could be separated by fractional distillation, they could be identified by these physical constants. In order to determine the approximate composition of the crude turpentine, the additive percentages of the combined fractions are plotted against the corrected boiling points of the fractions and also against their specific gravities. At that point that the boiling point curve increases and the specific gravity curve decreases the distillate is changing from pinene to dipentene.

RESULTS OF SEMI-COMMERCIAL TESTS.

Method of Plotting Data.

In order to show more clearly just what effect the different variables had on the extraction of the rosin from the wood by this process, it was decided to show the progress of the extraction graphically for each set of conditions. The following curves are plotted so that the ordinates show to percent of rosin recovered on the basis of the total rosin in the wood as determined by the control to be 100 percent. The abscissa are divided proportionally to the time that each solvent was in contact with the wood. On all the curves, 1/2 inch represents the time that one solvent was in contact with the wood in one retort. Thus solvent I was in only one retort and hence the rosin extracted by it is to be plotted unit distance from the origin which is 1/2 inch. Solvent II was in contact with the wood twice as long as solvent I and hence the interval between its location on the curve and the location of the point for solvent I will be two units or one inch. These curves then show the progress of the extraction throughout the entire process. Their slope is the rate of extraction and this seem to be of importance in some cases. Their final reading is the total percent of rosin extracted and is also the efficiency of extraction for that run.

The Effect of Time.

Table 3 shows the results of those runs which will illus-

trate the effect of time on extraction.

Table 3.

Run No.	Size of Chips	Pressure of Extraction	Time of Extraction	Rosin in Wood,%	Rosin Yield,%
1	3/16	0	15	29.92	72.60
2	3/16	0	45	25.10	75.00
3	3/16	30	15	23.00	91.6
4	3/16	30	45	25.92	93.7
11	5/8	30	15	12.90	68.5
12	5/8	30	45	12.23	68.9

The curves are shown in Figures 7, 8, and 9. In all cases, it must be remembered that the actual units of abscissae for the curves of runs 2, 4, and 12 are three times as large as the units of curves 1, 3, and 11. For the sake of comparison the curves were plotted in this manner.

In each case the effect of time is very slight. Under the most satisfactory conditions, namely with 3/16 inch chips and a pressure of 30 pounds, the efficiency is raised only 2.4 percent when the time is increased three times. It is probable that this figure would be made considerably smaller if the wood of run 3 had been as rich in rosin as that of run 4.

A peculiarity noticed in these curves is that with long time extractions, there is a much lower rate of extraction while the first two or three solvents are on the wood and a slightly greater rate of extraction for the remaining time of extraction. However, the essential result obtained is that greater time has

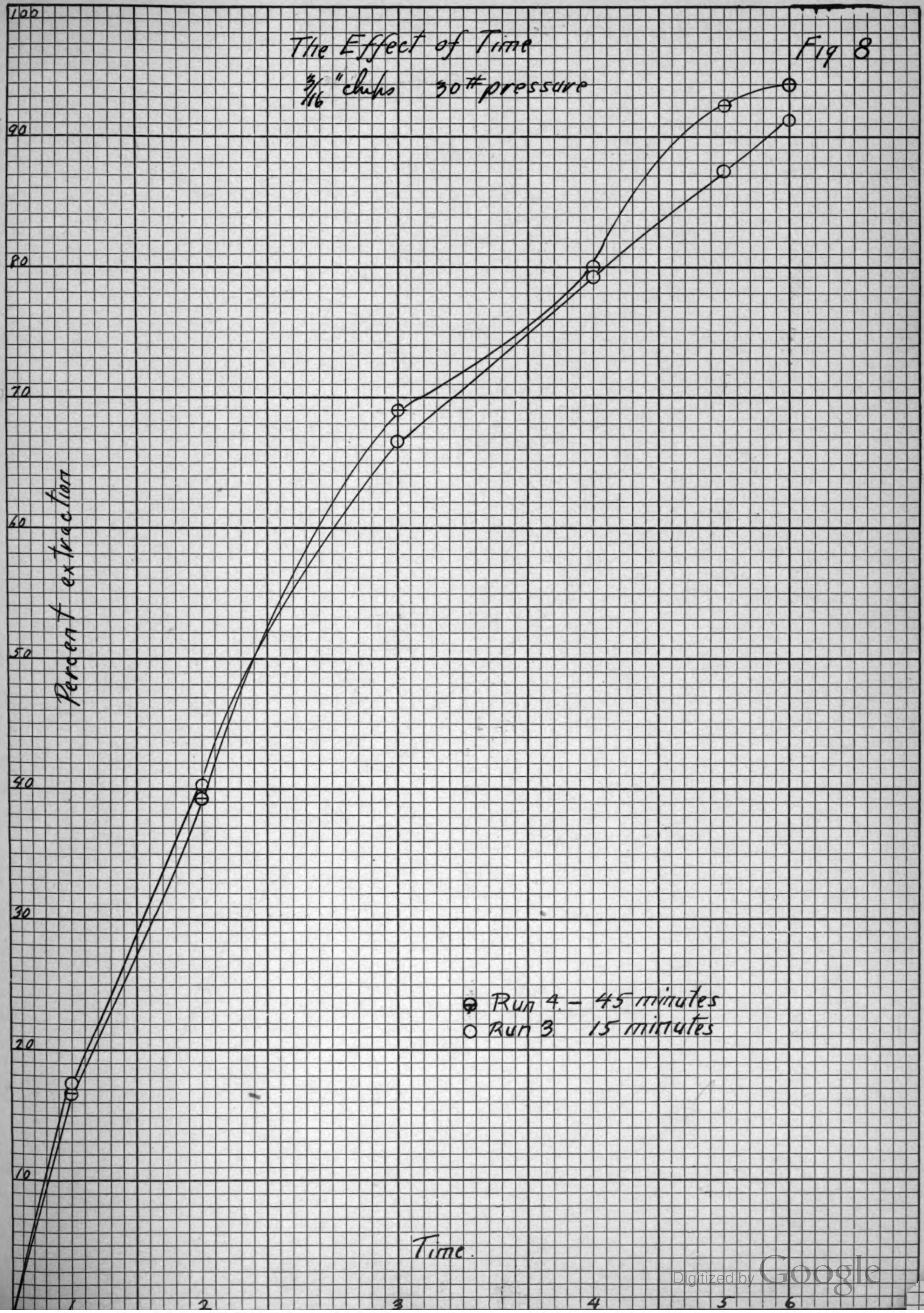
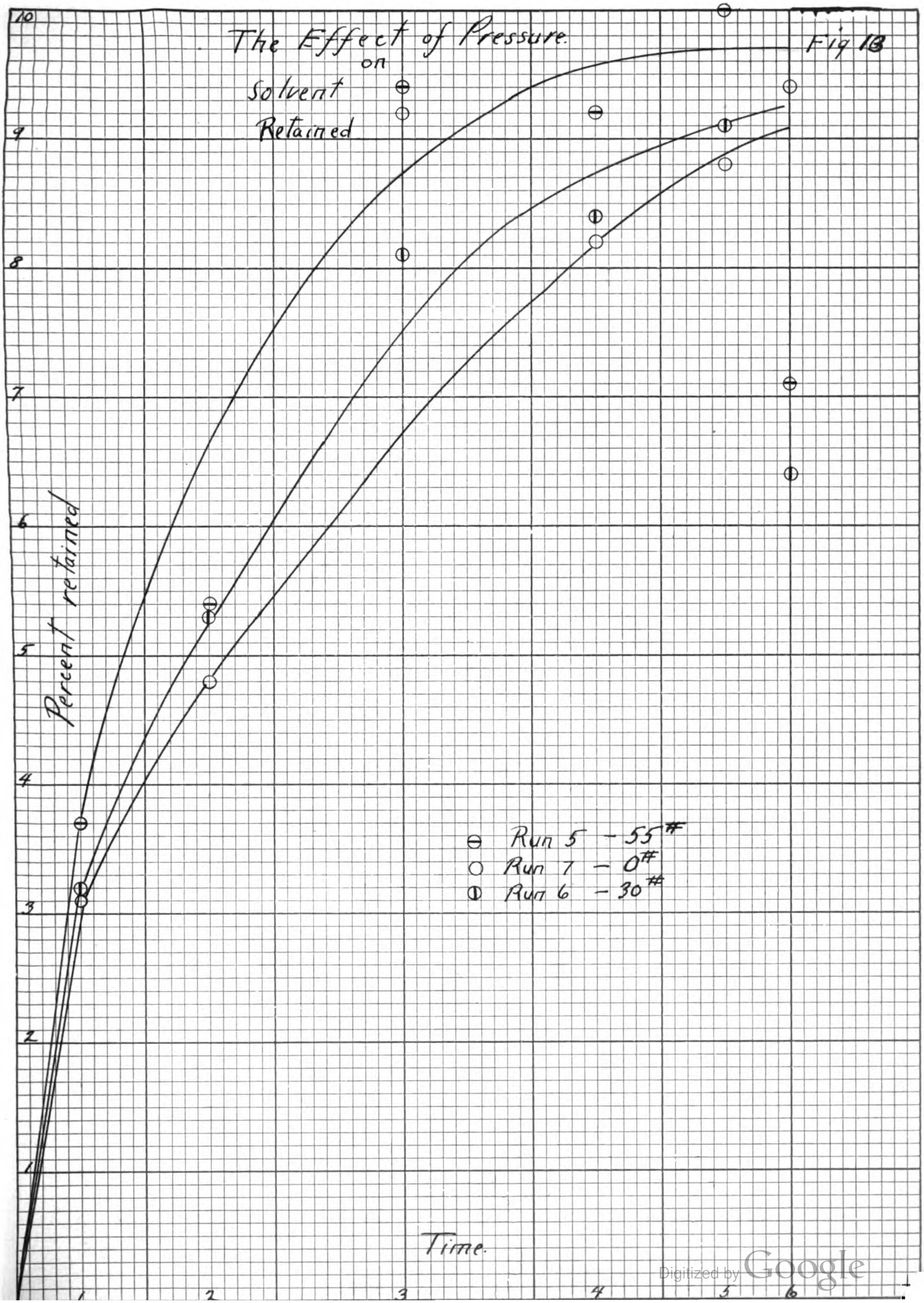


Fig 8

The Effect of Time

3/16" chips 30# pressure



The Effect of Pressure on Solvent Retained

Fig 13

pronounced for 30 pound extraction. However extraction at 55 pounds gives an efficiency that is between these other two conditions. This is not only true for the final results, but the curves show that throughout the extraction the percent of rosin being recovered was always lower than at 30 pounds pressure.

The curves of each set, and especially those of Fig. 10 show that the treatment by solvent I is practically the same in all cases and is probably only the washing off of the rosin on the surface of the chips. It is in the solvents which follow that the true effect of pressure on the extraction is observed. The slope of the curves for runs 1 and 7, which were carried out under atmospheric conditions, show that the solvent is very slow to penetrate the chips at first; but when the extraction is nearly completed, in solvents V and VI especially, the rate of extraction increases. It would be of interest to wash with another solvent in this case to determine whether this increased rate of extraction would continue and give an efficiency which would be more nearly equal to that obtained by pressure extractions.

The unexpected results of run 5, that extraction at 55 pounds was not as efficient as extraction at 30 pounds may be ex explained in the following manner:-

The curves of Fig. 13 show the percent of solvent retained in the chips under different pressure extractions. These curves show that, with increased pressure, the amount of solvent retained in the chips increases. It is also clearly illustrated

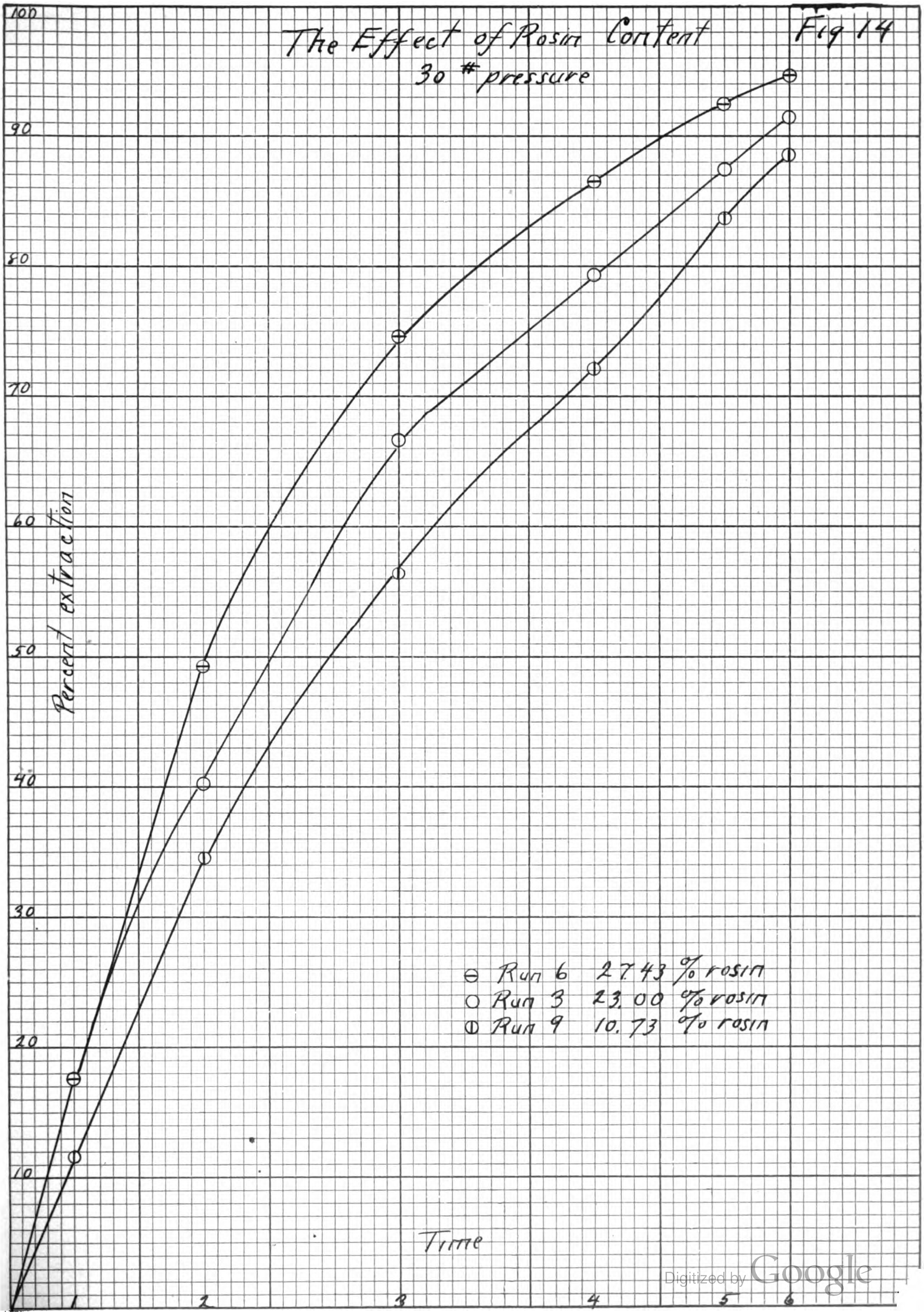
here that the solvent is slow to penetrate the chips when atmospheric extraction is employed as the curve for run 7 is still rising when the last solvent is removed. However, there seems to be nearly complete penetration when the third solvent has been removed in the pressure extractions. The line beyond here is probably horizontal for the 55 pound extraction and nearly so for the 30 pound extraction. Thus it may be that under a pressure of 55 pounds, the solvent is forced into the chips soon after the extraction begins and remains there so that no fresh solvent can come in contact with the rosin. It is also possible that the boiling is greatly reduced at 55 pounds pressure and hence there is less circulation of the solvent and less solvent action on the chips.

The Effect of Rosin Content.

Table 5

Run No.	Size of Chips	Pressure of Extraction	Time of Extraction	Rosin in Wood,%.	Rosin Yield,%.
9	3/16	30	15	10.73	88.8
3	3/16	30	15	23.00	91.6
6	3/16	30	15	27.43	94.8
7	3/16	0	15	13.32	70.8
1	3/16	0	15	29.92	72.60

The data of table 5 is plotted in the curves of Figures 14 and 15 and shows the effect of the rosin content of the wood on the the efficiency of extraction, all other things being equal. The results, as shown by both sets of curves, are that the


The Effect of Rosin Content
30 # pressure
Fig 14
Percent extraction
Time
100
90
80
70
60
50
40
30
20
10
1
2
3
4
5
6
⊖ Run 6 27.43 % rosin
○ Run 3 23.00 % rosin
⦶ Run 9 10.73 % rosin

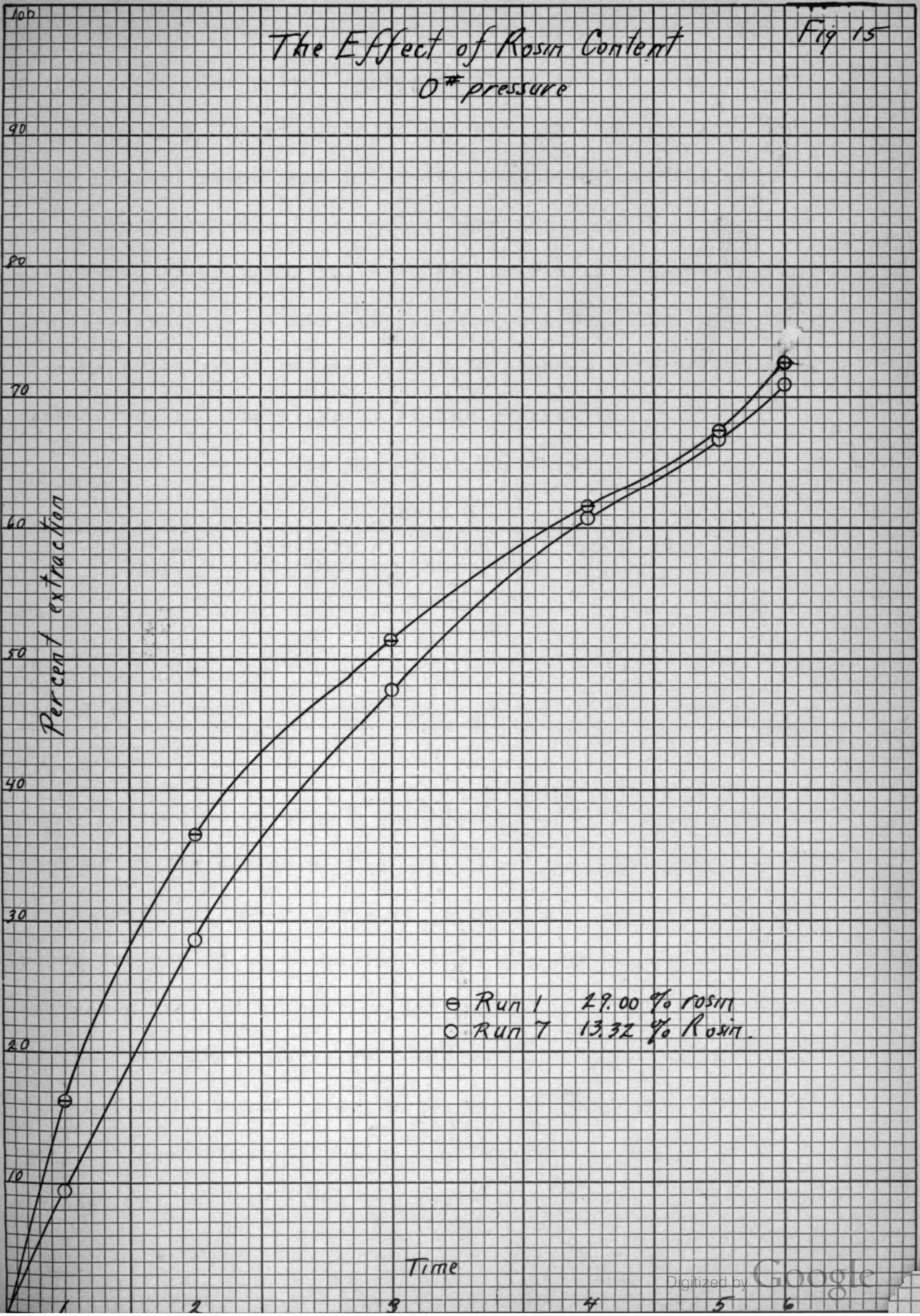


Fig 15

The Effect of Rosin Content

0# pressure

the richer wood gives higher efficiency of extraction than the poorer wood. The effect seems to be greater for pressure extractions than for atmospheric extractions. An increase of 15.68 percent rosin content of the wood increases the efficiency 4.5 percent over its lower value when atmospheric extractions are used. An increase of 16.70 percent rosin content increases the efficiency of extraction 6.75 percent over its lower value when the pressure of extraction is 30 pounds. The effect is evident throughout the progress of the extraction.

The Effect of Moisture.

Table 6.

Run No.	Size of Chips	Pressure of Extraction	Time of Exaraction	Rosin in Wood,%.	Rosin Yield,%.
9	3/16	30	15	10.73	88.8
10	3/16	30	15	10.70	72.1

The chips of run 9 were dry- those of run 10 were wet.

The results of runs 9 and 10 are shown in table 6 and are plotted in the curves of Fig. 16. They show that when moisture is in the wood, the extraction is retarded considerably. The curve for run 10 in which the chips were not dried shows a much decreased rate of extraction during the first two washings due to the fact, that the solvent had to replace the water in the wood before it could dissolve the rosin. After the second solvent had been removed, the rate of extraction was about as great as that for the dry chips but the retarding action of the first

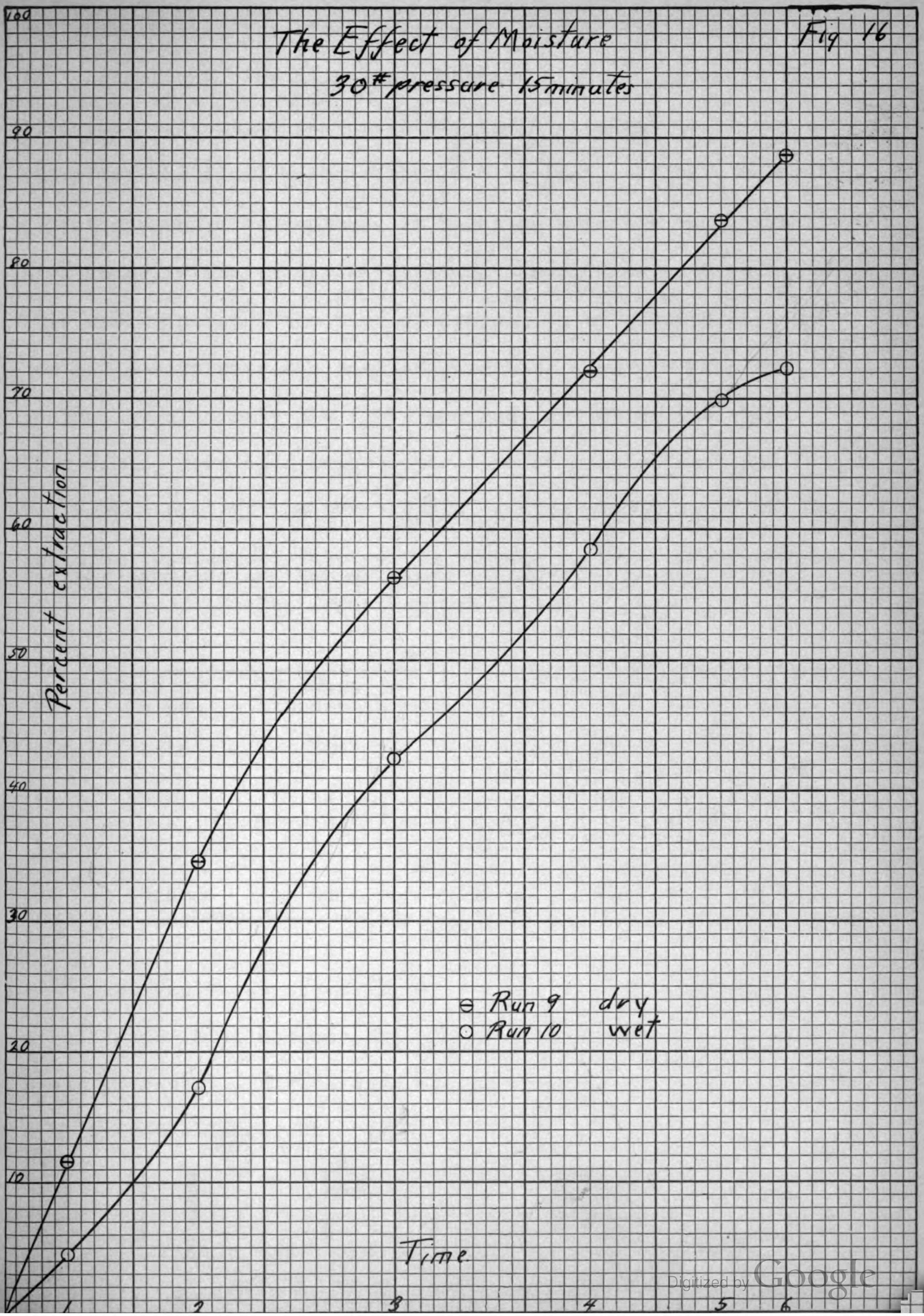


Fig 16

The Effect of Moisture

30# pressure 15 minutes

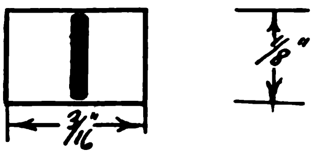
solvents lowered the efficiency by an amount equal to that retarding action. The difference between the two curves at the time that solvent II was drawn off was equivalent to 17 percent and after the final wash it was 16.5 percent. If this constant difference could be kept up, it might be possible to obtain as high an efficiency with wet chips by using a seventh wash. This would increase the time of extraction by about 8 percent but would eliminate the cost of operating a vacuum pump and would also eliminate the time required for drying the chips. This would more than balance the time of another wash and it would seem that if the curve would continue as is supposed, it would be considerably cheaper to operate in this way, unless the cost of handling another solvent would be too great.

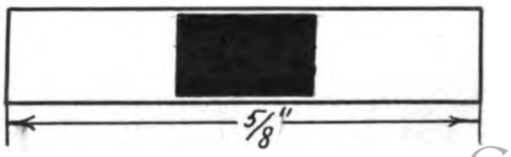
The Effect of the Size of Chips.

Table 7.

Run No.	Size of Chips	Pressure of Extraction	Time of Extraction	Rosin in Wood, %.	Rosin Yield, %.
9	3/16	30	15	10.73	88.8
11	5/8	30	15	12.90	68.5
4	3/16	30	45	25.92	93.7
12	5/8	30	45	12.50	68.9

The results as shown in table 7 and in the curves of figures 17 and 18 illustrate the effect of the size of chip on the yield of rosin. In each case the efficiency was lowered considerably by the larger chips.





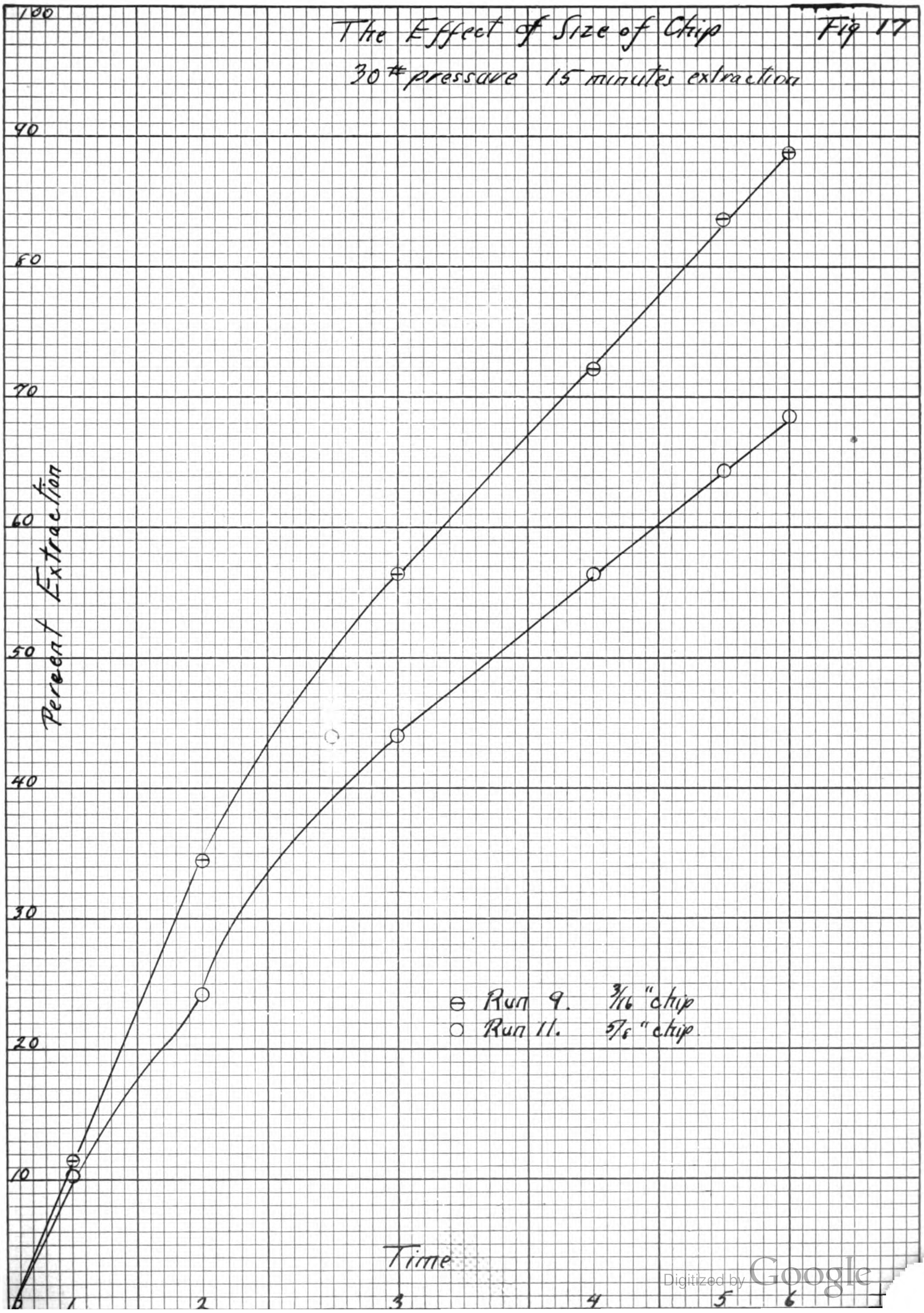


Fig 17

The Effect of Size of Chip

30# pressure 15 minutes extraction

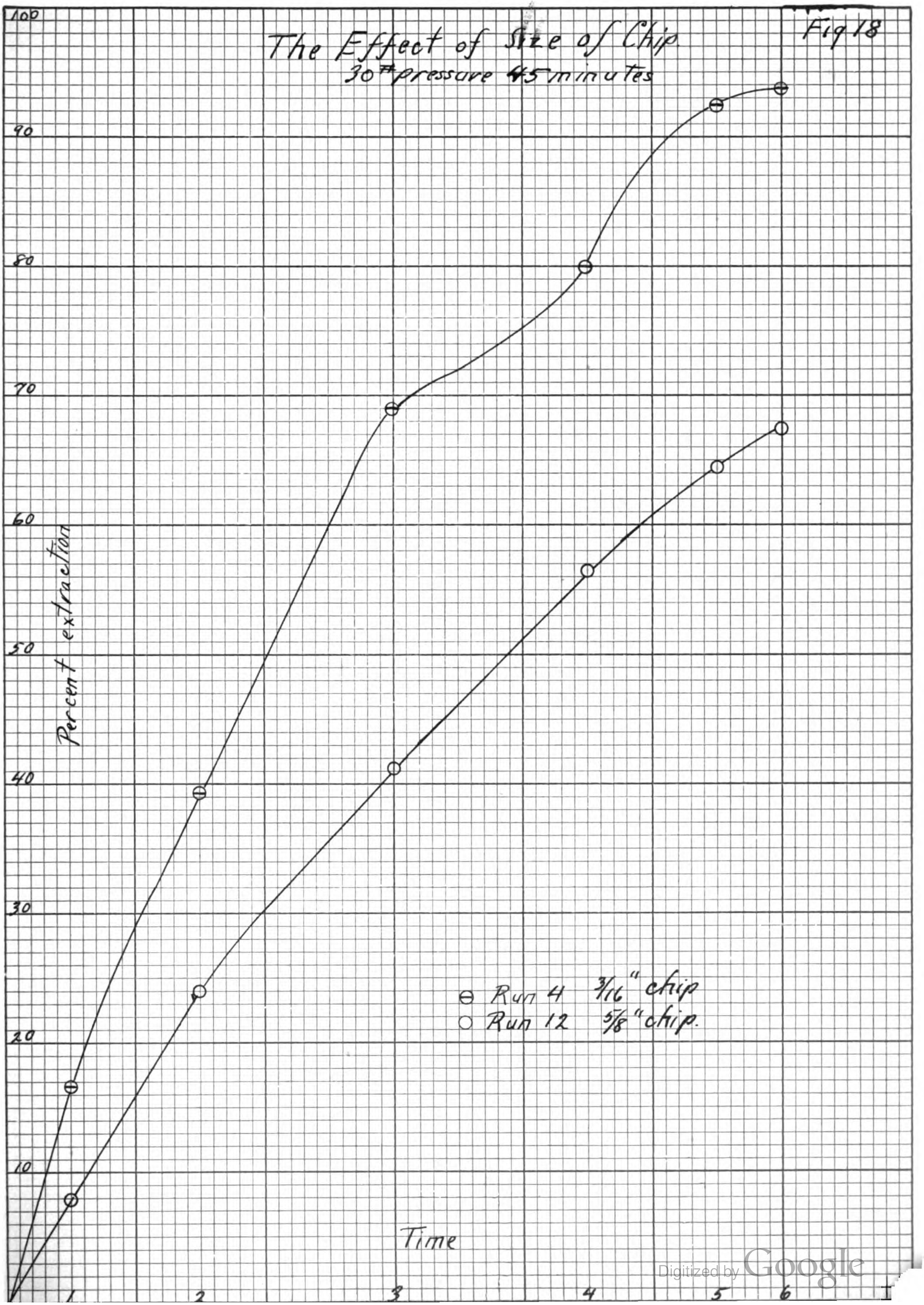


Fig 18

The Effect of Size of Chip

30# pressure 45 minutes

The sketch on the previous page shows the relative sizes of the chips used. The smaller ships averaged 3/16 x 1/8 x 1 inch, the length with the grain being 3/16 inch. The larger chips averaged 5/8 x 1/8 x 1 inch, the length with the grain being 5/8 inch. Hence in increasing the size of chips from the 3/16 to the 5/8 inch the volume is increased is increased from .023 cu. in. to.0 78 cu.in. or three times.

Most of the penetration of the solvent is with the grain of the wood which is the direction in which the resin ducts extend. Assuming that the penetration will proceed largely in the longitudinal direction,then with an efficiency of about 90 percent as was obtained with the small chips, it could be assumed that the solvent had penetrated 90 percent of the distance into the chip and that the remaining 10 percent was not touched by the solvent and was not extracted. This is represented by the shaded portion in the above sketch. With the larger chips under the same conditions,the solvent would penetrate at the same rate as before and would then extract the rosin from the same proportion of the wood as it had on the former chips. The unextracted portion would then be 10 percent for each volume equal to that of the smaller chips,but as there was three times the volume in the larger chips as in the smaller ones, the loss would be tripled and would then be 30 percent which was about what was obtained in these experiments.

It may be that the solvent cannot penetrate this inner portion of the chip, not because it has not had time enough, but

because the pressure of the air in the chip prevents its penetration. The volume of this air in the small chip becomes so small when the pressure is raised to 30 pounds, that a higher pressure has no further effect on it in all probability, but on the larger chips, a higher pressure might reduce its volume and in this way give more penetration with a resultant higher extraction of rosin. It would be of interest to try extractions on these larger chips using pressures of 50 to 60 pounds and see what the efficiency is under such conditions.

Solvent Loss.

Table 8

Run No.	Size of Chips	Pressure of Extraction	Time of Extraction	Solvent Loss,%.	Efficiency of extraction.
1	3/16	0	15		72.60
2	3/16	0	45	6.87	75.0
3	3/16	30	15	3.79	91.6
4	3/16	30	45	4.30	94.0
5	3/16	55	15	3.74	85.3
6	3/16	30	15	3.16	94.8
7	3/16	0	15	2.38	70.8
9	3/16	30	15	3.43	88.8
10	3/16	30	15	3.79	72.1
11	5/8	30	15	3.46	68.5
12	5/8	30	45	3.85	68.9

Table 8 is a summary of the solvent losses of the various runs. The results do not show very clearly the effect of the

different variables on the solvent loss. The apparatus was found not to be entirely suitable in its present form for this part of the study. However, some conclusions can be derived from this data. It seems that increased time and pressure will increase the solvent loss. Runs 7, 6, and 5 show the effect of pressure and runs 11 and 12 show the effect of time. The lowest of these solvent losses compares with the average loss in several of the larger commercial plants. It was found in several cases that solvent would collect in pockets in the apparatus and would appear after the test had been completed or when the next test was begun. It might be possible to get better results on solvent loss if the apparatus were run continually for several days, thus allowing no intervening time between tests. This would be more nearly comparable with commercial operation.

The curves of Fig. 13 show that the solvent retained in the chips is from 7 to 10 percent while in the laboratory tests, it was from 14 to 19 percent.

Crude Turpentine.

Only three analyses of the crude turpentines were made. The volatile oils from runs 1, 2, and 3 were combined and a sample taken. The analytical curves of this material are shown in Fig. 19 . The wood used in these runs was kept in bags for several weeks and much of the lighter portions of the turpentine evaporated. The curve shows a considerable amount of rosin oils and some solvent was noticeable also. The curves would give the approximate composition of this crude turpentine as:-

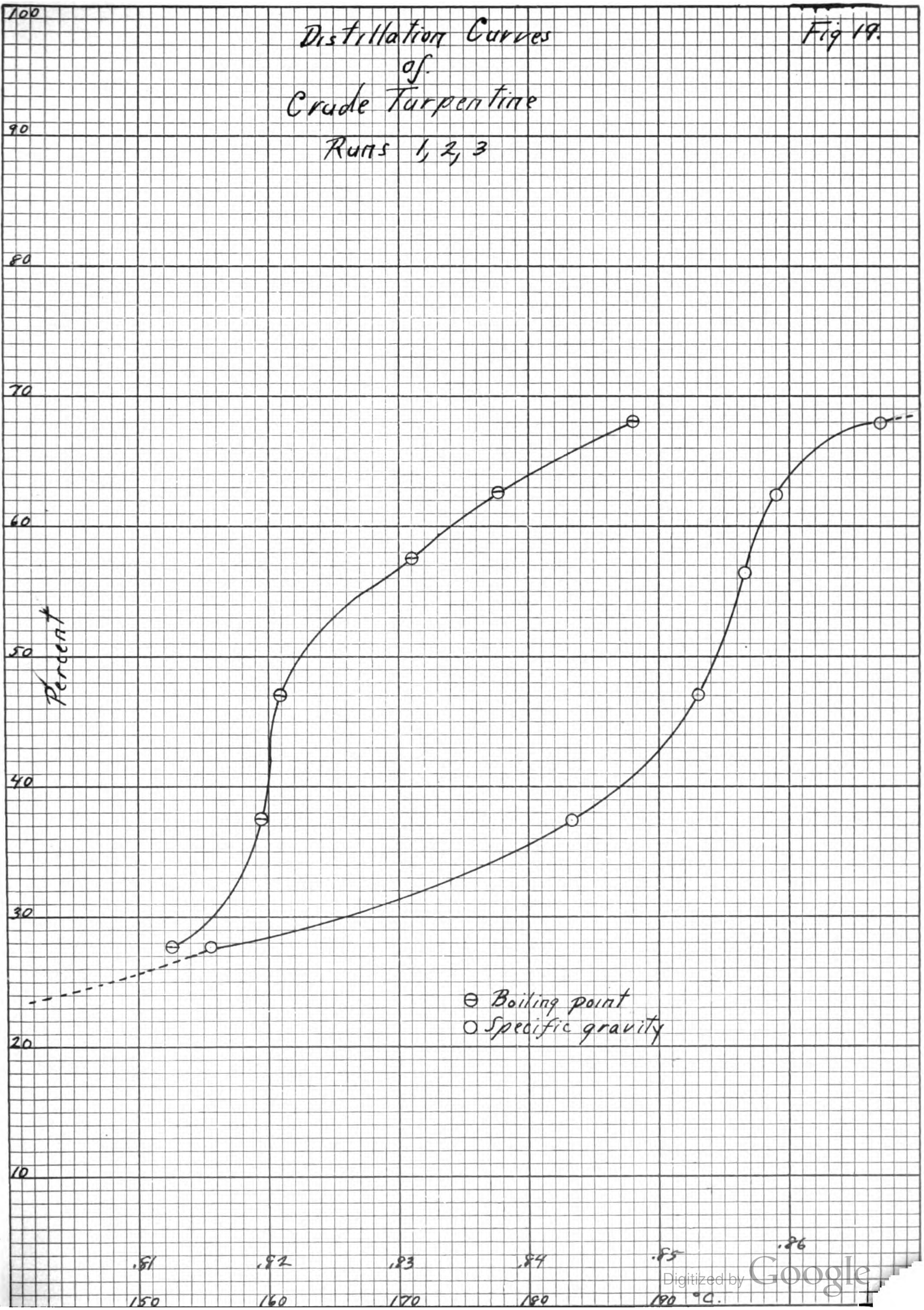


Fig 19.

Distillation Curves
of
Crude Turpentine
Runs 1, 2, 3

Solvent and Rosin Oil[b]	30
Turpentine	18
Pine oil	52

Figure 20 is the analytical curve of a sample of the combined crude turpentines from runs 4, 5, and 6. There is some solvent and rosin oil in this sample also, but not as much as in the first sample. The composition is about:-

Solvent and Rosin Oil[b]	18
Turpentine	54
Pine oil	28

This curve shows considerable amount of dipentene. This is not a normal constituent of gum turpentines and it seems to be a decomposition product of pinene which may be formed by distillation or may be a normal constituent of lightwood.[a]

Figure 21 shows the distillation curves for a sample of the crude turpentine from runs 9, 10, 11, and 12. It shows considerable solvent and rosin oils and about the same amount of dipentene in the turpentine as the previous sample. The analysis shows about:-

Solvent and Rosin Oil[b]	25
Turpentine	54
Pine oil	21

Solvent.

Figure 22 is a fractional distillation curve of the solvent used following its purification from rosin after the last run.

(a) Forest Service Bulletin 109 - Hawley and Palmer.

(b) The rosin oil was only a small percent of the total.

Fig 20

Distillation Curves of Crude Turpentine Runs 4, 5, 6.

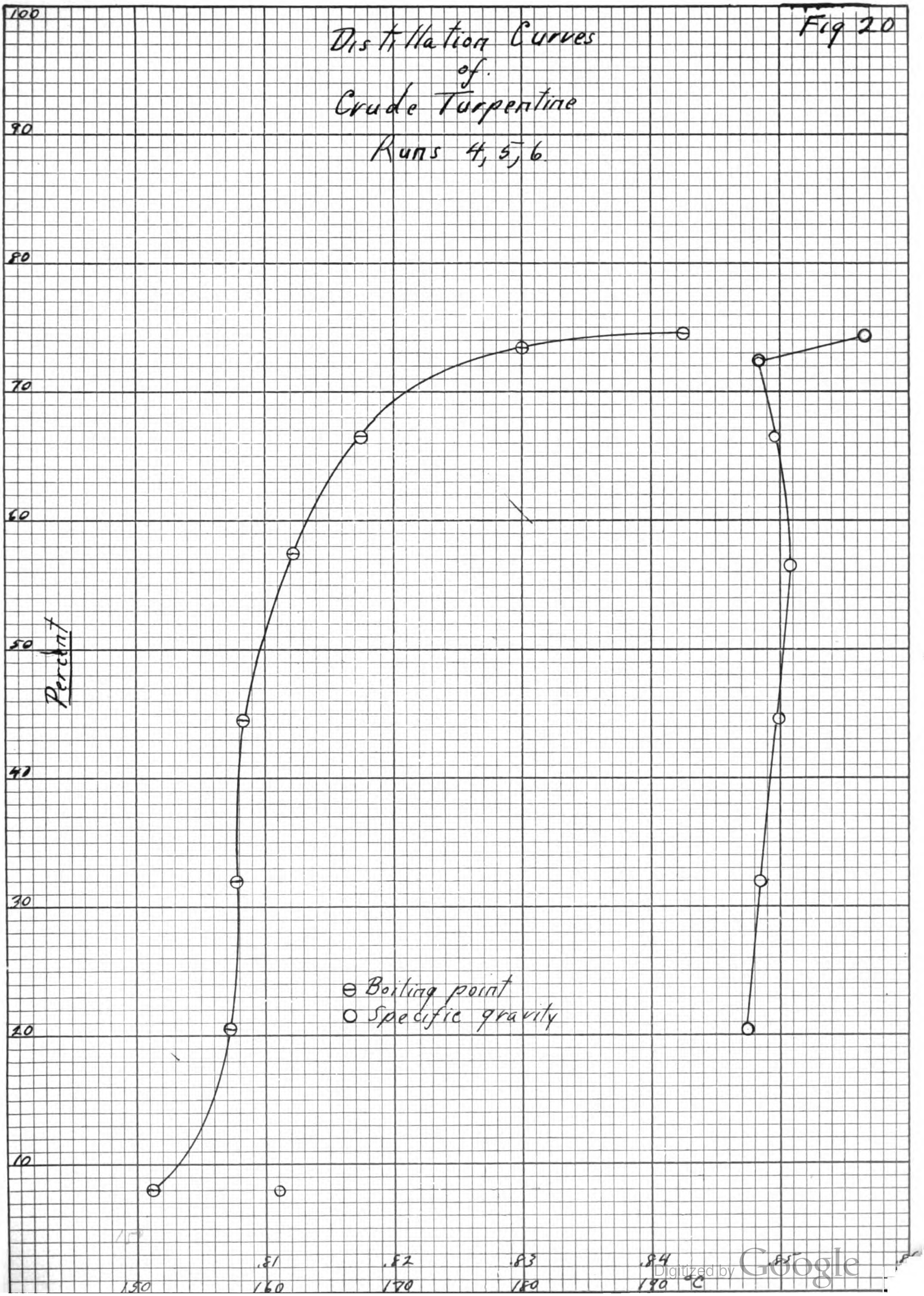
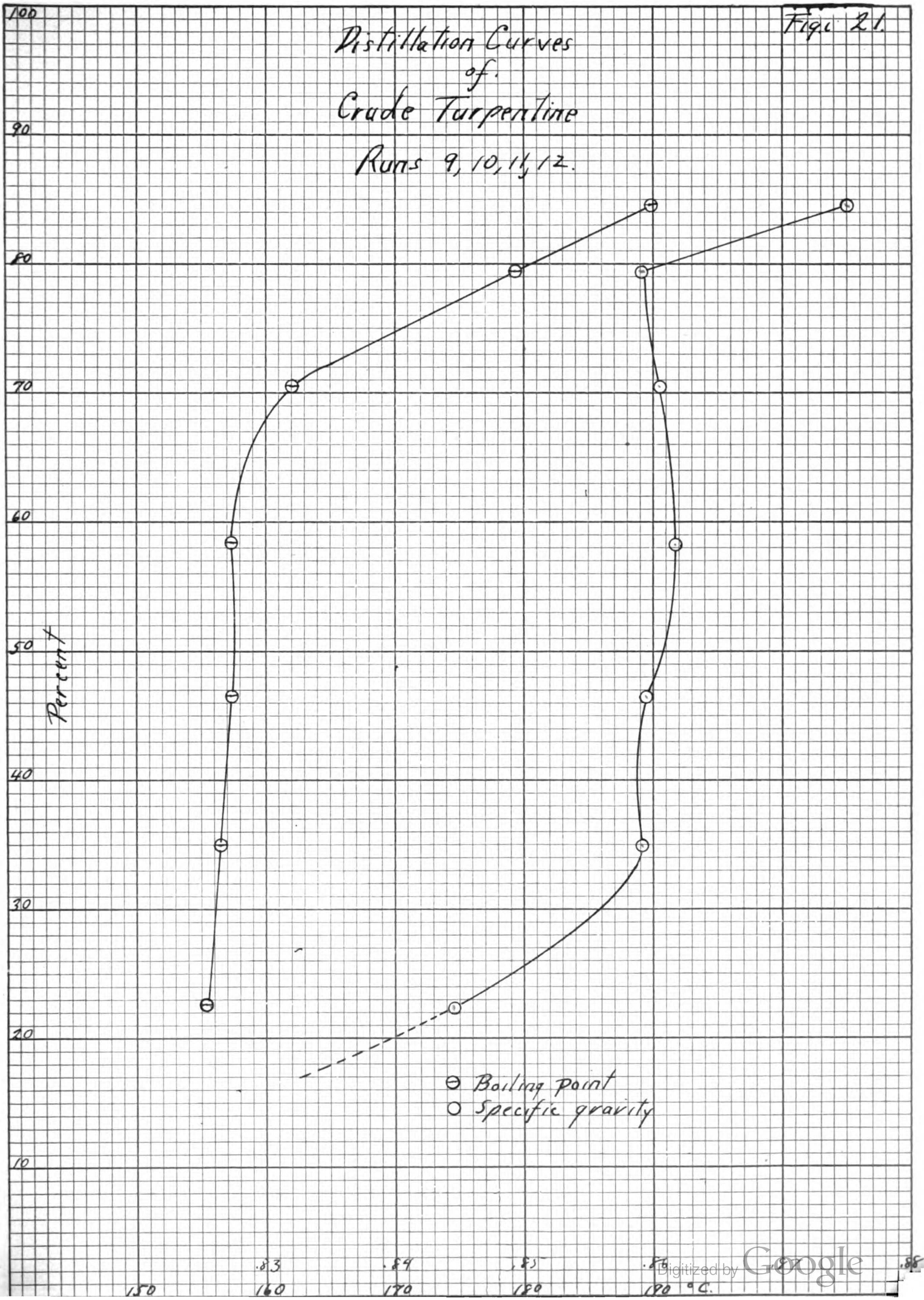
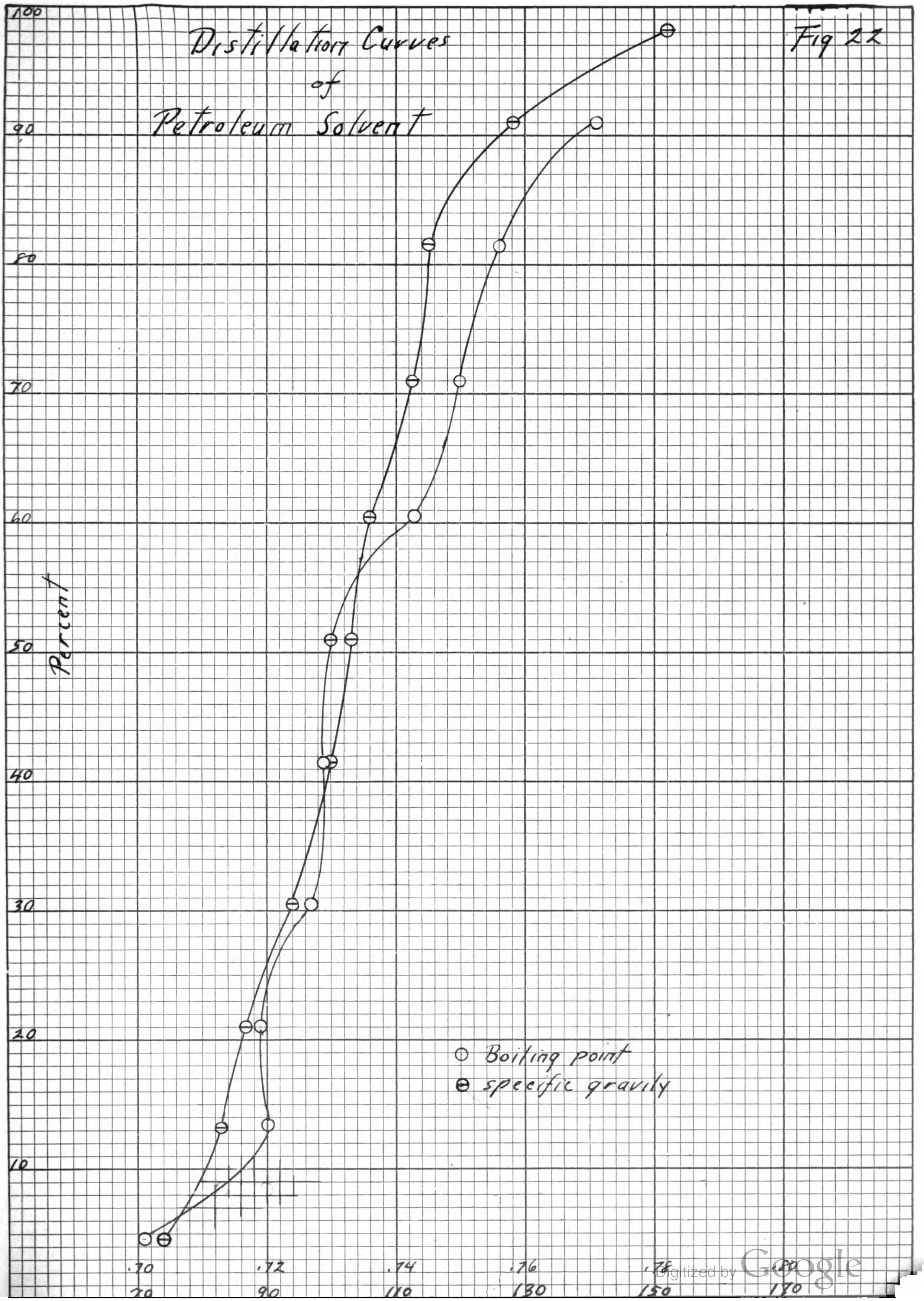


Fig. 21.

Distillation Curves
of
Crude Turpentine
Runs 9, 10, 11, 12.





Distillation Curves of Petroleum Solvent

Fig 22

The solvent used in these experiments was distilled in a column before it was used and all the material below a boiling point of 70 deg.C. was discarded. The curve shows that practically no material came over below 70 deg. C. and that 91 percent of it distilled below 141 deg. C. This, with the gravity, is evidence that there was little or no turpentine or heavier oils in the solvent even after it had been used for 12 runs.

The Formation of a Precipitate.

In a few of the runs, the fifth, sixth, and seventh especially, considerable amounts of a dark brown precipitate came out with the solvent. It was heavier than the solvent and lighter than water. Yaryan experienced this same trouble and calls it a pitchy rosin. He has taken out a patent for its removal.(a) At first, it was thought that this might be an emulsion of rosin and if this were the case, the amount of rosin should be determined and correction made. On exposure to the air for several days, the material became darker. It had a sticky feeling and was somewhat gritty. In order to determine what the cause of its formation was, a series of tests were made on it.

A portion of about 25 grams was heated in a vacuum in a distilling flask. Water and solvent distilled off below 100 deg C. and at about 140 deg. C there was very little sign of any melted material. The residue seemed to be hard and granular. Some of the fresh precipitate was dissolved in chloroform and filtered. A red brown residue was left on the filter. The solution seemed to have solid material in suspension. The residue

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(a) U.S.Pat. 934257

on the filter was completely soluble in hydrochloric acid to form a reddish yellow solution which when neutral was a deep red color. This solution showed a strong test for iron by the ferrocyanide test and also a slight test for copper.

Some of the fresh precipitate was dissolved in chloroform and without filtering, was extracted with hydrochloric acid. The water soluble material was separated out and when neutralized gave a red solution. Upon boiling, a flocculent precipitate was formed. This with the fact that the solution contained iron and was red was evidence that acetic acid was present. Another test for acetates was then made. A portion of this hydrochloric acid extract was treated with ethyl alcohol and allowed to stand over night. If any acetic acid were present, it would combine with the alcohol in the presence of the free hydrochloric acid and the ethyl acetate could be identified by its odor. The mixture was distilled and at about 80 deg. C. a few cubic centimeters of material distilled after which the boiling point jumped to 98 deg.C. Ethyl acetate boils at 78 degrees and as this first fraction had the distinctive odor of ethyl acetate, it was decided that there was acetic acid in this precipitate. The residue from the acid extract, when evaporated, gave a thin film of a black gum or rosin. Similiar extractions were made with sulphuric acid and iron and acetic acid determined qualitatively as before.

The conclusions arrived at were that this precipitate was not introducing any error into the calculations as it did

not contain any appreciable amount of rosin. It is a well known fact that the acetate of iron, upon boiling will hydrolyze and form an insoluble basic salt. It has also been proven that as lightwood becomes older, the amount of acetic acid in it increases. From this, it can be concluded that this acetic acid in the wood corroded the iron retorts and upon boiling the solution, the insoluble basic ferric acetate was precipitated out. This also occluded some of the rosin. It may be that this rosin is that part of common rosin which is insoluble in certain solvents, especially petroleum distillates, as the dark chloroform solutions could be completely cleared up by adding an excess of the solvent used in these experiments.(a) There was also some sawdust, sand, and charred wood in this precipitate.

(a) Collophony - Commercial Organic Analysis. Allen

See foot notes especially.

CONCLUSIONS AND APPLICATION.

All of the plants which are recovering ~~recovering~~ rosin by the extraction process are operating under atmospheric pressure. Hence, in order to get satisfactory results, it is necessary for them to carry out the extraction for a much greater length of time than any of these experiments required. It is known that in commercial plants the wood used is shredded after being chipped. This means that there is very little use that can be made of the wood after extraction excepting for fuel. The shredded wood is not satisfactory for pulp manufacture although there is a possibility that the extracted wood in this form can be used for the production of ethyl alcohol.

The process used in these experiments is slightly different from any used at the present time in that the apparatus was built with a reflux condenser system. This allowed all the solvent that had distilled off to return to that unit from which it had vaporized. It was this refluxing system that made pressure extractions possible.

The wood used in these tests was passed through an ordinary pulp wood chipper and was not subsequently shredded as is done in the commercial plants. This would then reduce the cost of reducing the wood considerably, as there would be no need for a shredder and the power to operate it would be saved.

The 5/8 inch chips used in runs 11 and 12 are sufficiently large for the production of pulp and paper, and, if this size

could be used with satisfactory extraction results, there would be considerable demand for the extracted chips as a scource of pulp. However it is probable, that the 3/16 inch chips would be too small for the production of a good grade of strong paper. As was stated before extractions on the 5/8 inch chips might be made more efficient if higher pressures were used, but it was found that with a pressure of 55 pounds, the wood was darkened considerably. This might have a bad effect on the pulp and would lower its grade.

A method which might be used to advantage if these larger chips cannot be extracted more thoroughly, would include this process followed by the Whitaker and Bates method.(a) Thus 65 to 70 percent of the rosin could be removed in the extraction process as rosin while the remainder could be removed by caustic soda as the wood was being prepared for pulp. This would then free the wood from all the resinous material.

The richness of the wood is going to affect the operation of the process considerably. Not only will the poorer wood contain less rosin in the first place, but it will give lower yields with respect to the rosin contained, than will the richer wood. This is clearly shown in table 5 and the curves of that data. A plant cannot select the richest wood and discard the wood that is not as rich in oleoresin and hence this effect will have to be taken into account in the utilization of the wood

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(a) See page 3 - Chemical Utilization of Southern Pine Waste.

Whitaker and Bates. Jour. Ind. and Eng. Chem. Apr. 1914

after it has been extracted. While it is possible to get 94.8 percent of the rosin out of the wood when its rosin content is 27.43 percent, it is only possible to get 88.8 percent of the rosin out when the rosin content is only 10.73 percent, all other conditions of extraction being equal.

The work included in these experiments was carried out in co-operation with the Forest Products Laboratory in the Department of Wood Distillation and under the direction of Mr. R.C.Palmer, Chemist in Forest Products, U.S. Forest Service.

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United States Patents. No.922369 Varyan
918990 Frankforter
934257 Varyan

APPENDIX.

DATA ON LABORATORY AND SEMI-COMMERCIAL TESTS.

DATA OF LABORATORY EXTRACTIONS.

Test Number	1&2	3	4
Time of extraction, min.	10	20	10
Time of steaming, min.	60	60	90
Specific gravity of solvent	0.7166	0.7281	0.7270
Sp.gr. of steam distillate	0.7270	0.7266	0.7284
Sp.gr. of solv. with rosin	0.7510	0.7546	0.7552
Sp.gr. of distillate	0.7270	0.7266	0.7307
Weight of wood,gms.	670.5	300.0	300.0
Rosin extracted,gms,	142.96	83.91	61.50
Total solvent used,c.c.	3300	1995	2040
From previous test		945	990
Fresh solvent	3300	1050	1050
Collected by coil	59.6	7.0	13.0
Solvent drained off	2454	1705	1810
Distilled for rosin	1509	665	582
Distilled from rosin	1346	575	500
Steam distillate	605	295	280
Solvent reclaimed	2955.6	1917	2021
Solvent lost	345	78	19

RUN 1. Feb 8-12 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 30 pounds pressure for 90 minutes.

Vacuum distillation at 20 inches vacuum for 90 minutes.

Extraction at 0 pounds pressure for 15 minutes.

Recovery of solvent.

Steam distillation at 30 pounds pressure for 90 minutes.

Vacuum distillation at 20 inches vacuum for 90 minutes.

CHARGE. 3/16 inch chips.

Retort No.	Wt. of charge.
1	25.84 pounds.
2	26.30 " "
3	24.75 " "
Total	76.84 " "

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort.	Steaming.	Vacuum.	Total.
1	325 c.c.	0	325 c.c.
2	450	0	450
3	270	0	270

Specific gravity of turpentine - 0.8630.

EXTRACTION. (All data in gallons.)

Solvent.	I	II	III	IV	V	VI
In.	20.37	16.00	16.25	15.25	15.00	15.37
Out	18.37	14.12	15.25	15.00	15.37	16.50

6.37 gals. of solvent I were rosin free.-Spilled 0.37 gals

SOLVENT RECOVERY. (in gallons.)

Retort.	Steaming.	Vacuum.	Total.
1	2.42	0.28	2.70
2	2.31	0.0	2.31
3	1.25	0.0	1.25
Total			6.36

ROSIN RECOVERY.

Solvent distilled	47.74 gals.
Rosin recovered.	not weighed.

ANALYSIS OF FRESH WOOD.

Moisture determination.

Retort No.	1	2	3
Sample,gms.	10.1871	13.8685	12.5762
Water,c.c.	0.6	0.9	0.8

Control extraction.

Sample,gms.	11.824
Dry extracted sample.	7.511
Rosin by weight	3.330

SOLVENT ANALYSIS. (Grams rosin per 200 c.c. of solution.)

Solvent	I	II	III	IV	V	VI
In	3.25	2.30	1.80	1.00	0.00	0.00
Out	7.25	8.80	7.05	5.90	2.47	2.65

RUN 2. Feb 15-18 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 30 pounds pressure for 90 minutes.

Vacuum distillation at 20 inches vacuum for 90 minutes.

Extraction at 0 pounds pressure for 45 minutes.

Recovery of solvent.

Steam distillation at 30 pounds pressure for 90 minutes.

Vacuum distillation at 20 inches vacuum for 90 minutes.

CHARGE. 3/16 inch chips.

Retort No.	Wt. of charge.
1	25.35 pounds
2	25.05 " "
3	24.75 " "
Total	75.15 " "

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort	Steaming	Vacuum	Total
1	240 c.c.	0	240 c.c.
2	215	0	215
3	180	0	180

Specific gravity of turpentine - 0.8635.

EXTRACTION. (All data in pounds)

Solvent.	I	II	III	IV	V	VI
In.	92.03	94.48	96.43	90.77	92.05	94.07
Out.	71.45	82.44	79.93	86.35	89.35	92.23

SOLVENT RECOVERY. (In pounds)

Retort	Steaming	Vacuum	Total
1	11.22	0.14	11.36
2	12.82	0.0	12.82
3	10.13	0.0	10.13

ROSIN RECOVERY.

Solvent distilled	218.12 pounds
Rosin recovered	11.37 " "

ANALYSIS OF FRESH WOOD.

Moisture determination.

Retort No.	1	2	3
Sample,gms.	9.4600	10.6100	12.4240
Water,c.c.	7.5	8.0	7.25

Control extraction.

Sample,gms.	8.6070
Dry extracted sample.	5.6780
Rosin by weight	1.9900

SOLVENT ANALYSIS. (Grams of rosin per 200 grams of solution.)

Solvent	I	II	III	IV	V	VI
In.	5.90	2.47	2.65	0	0	0
Out.	12.55	12.95	11.45	4.15	2.90	1.55

RUN 3. Feb 23-26 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 30 pounds pressure for 60 minutes.

Vacuum distillation at 20 inches vacuum for 60 minutes.

Extraction at 30 pounds pressure for 15 minutes.

Recovery of solvent.

Steam distillation at 30 pounds pressure for 90 minutes.

Vacuum distillation at 20 inches vacuum for 90 minutes.

CHARGE. 3/16 inch chips.

Retort No.	Wt.of charge.
1	25.45 pounds.
2	23.85 " "
3	24.10 " "
Total	73.40 " "

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort.	Steaming	Vacuum	Total
1	210 c.c.	0	210 c.c.
2	115	0	115
3	285		285

Specific gravity of turpentine - 0.8635

EXTRACTION. (All data in pounds.)

Solvent	I	II	III	IV	V	VI
In	89.28	89.07	91.39	87.85	89.48	92.16
Out.	75.68	78.24	78.10	82.90	95.14	91.95
Sludge	2.90		1.80			

Analytical samples from solvents - 1.4 pounds.

SOLVENT RECOVERY. (In pounds.)

Retort.	Steaming.	Vacuum.	Total.
1	13.78	0	13.78
2	7.72	0	7.72
3	14.92	0	14.92

ROSIN RECOVERY.

Solvent distilled	210.66	pounds.
Ice	1.87	" "
Rosin recovered	12.07	" "

ANALYSIS OD FRESH WOOD.

Moisture determination.

Retort No.	1	2	3
Sample,gms.	11.5590	15.2030	17.3225
Water,gms.	0.80	0.65	0.70

Control extraction.

Sample,gms.	15.6893
Dry extracted sample.	10.9206
Rosin by weight	3.4260

SOLVENT ANALYSIS. (Grams rosin per 200 gms of solution.)

Solvent.	I	II	III	IV	V	VI
In	4.15	2.90	1.55	0.00	0.00	0.00
Out.	12.75	12.45	12.85	5.25	2.70	1.36

NOTE: 3.16 pounds (estimated) of solvent was lost.

Turpentine from retort 3 seemed to contain solvent.

RUN 4. March 1-6 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 30 pounds pressure for 90 minutes.

Vacuum distillation at 20 inches vacuum for 90 minutes.

Extraction at 30 pounds pressure for 45 minutes.

Recovery of solvent.

Steam distillation at 30 pounds pressure for 60 minutes.

Vacuum distillation at 20 inches vacuum for 60 minutes.

CHARGE. 3/16 inch chips.

Retort No.	Wt of charge.
1	31.87 pounds.
2	31.06 " "
3	33.35 " "
Total	96.28 " "

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort	Steaming	Vacuum	Total
1	460 c.c.	10 c.c.	470 c.c.
2	520	10	530
3	610	0	610

EXTRACTION. (All data in pounds).

Solvent	I	II	III	IV	V	VI
In	87.58	93.42	90.79	89.86	85.40	92.45
Out	73.98	90.89	85.90	88.76	82.33	89.67
Sludge	1.24	2.45	0.72	0.23		

Analytical samples - 2.14 pounds

SOLVENT RECOVERY. (In pounds.)

Retort	Steaming	Vacuum	Total
1	7.66	0	7.66
2	9.91	0	9.91
3	12.46	0	12.46

ROSIN RECOVERY.

Solvent distilled	225.42 pounds.	
Rosin recovered	16.93 " "	

ANALYSIS OF FRESH WOOD.

Moisture determination

Retort No.	1	2	3
Sample, gms.	19.7180	18.7440	14.6845
Water, c.c.	2.30	2.20	1.40

Control extraction.(a)

Sample, gms.	14.9810
Dry extracted sample.	8.3101
Rosin by weight	4.1878

SOLVENT ANALYSIS. (Grams rosin per 200 grams of solution.)

Solvent	I	II	III	IV	V	VI
In	5.25	2.70	1.36	0.00	0.00	0.00
Out	16.90	14.75	18.40	5.90	2.50	0.70

(a). The results of the control indicated an error in sampling and another control extraction indicated 25.92 percent rosin.

RUN 5. March 5-9 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 30 pounds pressure for 90 minutes.

Vacuum distillation at 20 inches vacuum for 90 minutes.

Extraction at 55 pounds pressure for 15 minutes.

Recovery of solvent.

Steam distillation at 30 pounds pressure for 60 minutes.

Vacuum distillation at 20 inches vacuum for 90 minutes.

CHARGE. 3/16 inch chips.

Retort No.	Wt. of charge
1	33.69 pounds.
2	32.65 " "
3	32.10 " "
Total	98.44 " "

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort	Steaming	Vacuum	Total
1	500 c.c.	0	500 c.c.
2	600	0	600
3	675	10 c.c.	685

Specific gravity of turpentine - 0.8680

EXTRACTION. (All data in pounds.)

Solvent	I	II	III	IV	V	VI
In	86.94	81.00	88.10	69.48	73.86	78.11
Out	72.05	79.57	76.17	73.18	71.00	92.19

Samples - 1.45 pounds; Sludge 0.98 pounds

SOLVENT RECOVERY. (In pounds)

Retort	Steaming	Vacuum	Total
1	7.55	0	7.55
2	4.53	0	4.53
3	6.67	0	6.67

All retorts were cold and had been drained before steaming.

ROSIN RECOVERY.

Solvent distilled	200.42 pounds
Rosin recovered	not weighed.

ANALYSIS OF FRESH WOOD.

Moisture determination.

Retort No.	1	2	3
Sample, gms.	15.6984	15.9271	20.8918
Water, c.c.	1.50	1.55	2.05

Control extraction.

Sample, gms.	15.1469
Dry extracted sample.	8.9989
Rosin by weight	4.0185

SOLVENT ANALYSIS. (Grams rosin per 200 grams solution.)

Solvent	I	II	III	IV	V	VI
In	5.90	2.50	0.70	0.00	0.00	0.00
Out	20.05	18.10	19.95	7.92	2.42	1.05

NOTE: 3 pounds (estimated) of solvent was spilled.

RUN 6. March 12-16 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 30 pounds pressure for 90 minutes.

Vacuum distillation at 20 inches vacuum for 90 minutes.

Extraction at 30 pounds pressure for 15 minutes.

Recovery of solvent.

Steam distillation at 30 pounds pressure for 60 minutes.

Vacuum distillation at 20 inches vacuum for 90 minutes.

CHARGE. 3/16 inch chips.

Retort No.	Wt. of charge.
1	31.85 pounds.
2	30.56 " "
3	26.60 " "
Total	89.01 " "

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort	Steaming	Vacuum	Total
1	280 c.c.	0	280 c.c.
2	465	0	465
3	270	0	270

Specific gravity of turpentine - 0.8767

EXTRACTION. (All data in pounds.)

Solvent	I	II	III	IV	V	VI
In	82.10	78.79	90.92	74.26	83.78	81.76
Out	71.19	70.62	74.92	81.50	81.90	79.13

Analytical samples - 2.15 pounds; Sludge - 1.06 pounds.

SOLVENT RECOVERY. (In pounds.)

Retort	Steaming	Vacuum	Total
1	7.61	0	7.61
2	10.52	0	10.52
3	12.56	0	12.56

The retorts 2 and 3 were not drained before steaming.

ROSIN RECOVERY.

Solvent distilled	193.15 pounds.
Rosin recovered	

ANALYSIS OF FRESH WOOD.

Moisture determination.

Retort No.	1	2	3
Sample,gms.	15.1572	14.6640	13.5533
Water,c.c.	1.90	1.85	1.45

Control extraction.

Sample	13.4784
Dry extracted sample	8.0320
Rosin by weight	3.2551

SOLVENT ANALYSIS. (Grams rosin per 200 grams of solution.)

Solvent	I	II	III	IV	V	VI
In	7.92	2.42	1.05	0.00	0.00	0.00
Out	19.35	19.20	15.75	6.23	3.05	1.10

NOTE:- 8.50 pounds (estimated) solvent spilled.

RUN 7. March 24-27 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 30 pounds pressure for 75 minutes.

Vacuum distillation at 20 inches vacuum for 150 minutes.

Extraction at 0 pounds pressure for 15 minutes.

Recovery of solvent.

Steam distillation at 30 pounds pressure for 120 minutes.

Vacuum distillation at 20 inches vacuum for 120 minutes.

CHARGE. 3/16 inch chips.

Retort No.	Wt. of charge.
1	39.13 pounds.
2	34.94 " "
3	35.51 " "
Total	109.58 " "

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort	Steaming	Vacuum	Total.
1	225 c.c.	o	225 c.c.
2	150	10	160
3	lost	0	----

EXTRACTION. (All data in pounds.)

Solvent	I	II	III	IV	V	VI
In	80.46	79.38	77.70	80.02	72.80	78.59
Out	65.42	71.20	67.20	79.65	70.15	80.24

Samples for analysis - 0.75 pounds.

SOLVENT RECOVERY. (In pounds.)

Retort	Steaming	Vacuum	Total
1	9.86	0	9.86
2	6.30	0	6.30
3	5.14	0	5.14

Before steaming, 11.34 pound was drained and added to solv. VI.

ROSIN RECOVERY.

Solvent distilled	192.54 pounds.
Rosin recovered	10.89 " "

ANALYSIS OF FRESH WOOD.

Moisture determination

Retort No.	1	2	3
Sample, gms.	14.6728	13.0436	16.0764
Water, c.c.	4.40	3.80	5.10

Control extraction.

Sample, gms.	18.3167
Dry extracted sample.	11.0656
Rosin by weight	1.7514

SOLVENT ANALYSIS. (Grams rosin per 200 grams solution.)

Solvent	I	II	III	IV	V	VI
In	6.23	3.05	1.10	0.00	0.00	0.00
Out	10.70	8.90	7.05	3.40	1.71	0.92

Note: The moisture content of the sample for control extraction was 28.2 percent.

RUN 9. April 13-16 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 30 pounds pressure for 60 minutes.

Vacuum distillation at 20 inches vacuum for 150 minutes.

Extraction at 30 pounds pressure for 15 minutes.

Recovery of solvent.

Steam distillation at 30 pounds pressure for 60 minutes.

Vacuum distillation at 20 inches vacuum for 240 minutes.

CHARGE. 3/16 inch chips.

Retort No.	Wt. of charge.
1	37.27 pounds
2	37.28 " "
3	37.77 " "
Total	112.32 " "

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort	Steaming	Vacuum	Total
1	185 c.c.	0	185 c.c.
2	100	0	100
3	95	0	95

EXTRACTION. (ALL data in pounds.)

Solvent	I	II	III	IV	V	VI
In	78.93	78.66	93.45	75.22	81.78	83.95
Out	61.62	70.42	83.60	77.33	75.13	99.60

10.97 pounds of solvent VI was drained before steaming.

SOLVENT RECOVERY. (In pounds.)

Retort	Steaming	Vacuum	Total
1	5.62	0	5.62
2	3.44	0	3.44
3	7.65	0	7.65

All retorts were drained before steaming.

ROSIN RECOVERY.

Solvent distilled	206.64 pounds.
Rosin recovered	

ANALYSIS OF FRESH WOOD.

Moisture determination

Retort No.	1	2	3
Sample, gms.	18.3127	20.4499	17.9180
Water, c.c.	5.20	5.55	4.90

Control extraction.

Sample, gms.	18.7660
Dry extracted sample.	11.8230
Rosin by weight	1.4562

SOLVENT ANALYSIS. (Grams rosin per 200 grams solution.)

Solvent	I	II	III	IV	V	VI
In	4.20	1.95	0.69	0.00	0.00	0.00
Out	8.00	7.70	5.52	3.55	2.70	0.85

RUN 10. April 19-24 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 30 pounds pressure for 60 minutes.

No vacuum was drawn.

Extraction at 30 pounds pressure for 15 minutes.

Recovery of solvent.

Steam distillation at 30 pounds pressure for 60 minutes.

Vacuum distillation at 20 inches vacuum for 120 minutes.

CHARGE. 3/16 inch chips.

Retort No.	Wt. of chips.
1	37.66 pounds.
2	37.45 " "
3	37.73 " "
Total	112.84 " "

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort	Steaming	Vacuum	Total
1	75 c.c.	0	75 c.c.
2	75	0	75
3	120	0	120

EXTRACTION. (All data in pounds.)

Solvent	I	II	III	IV	V	VI
In	76.40	74.09	97.93	82.41	77.71	77.35
Out	61.48	65.43	83.79	80.31	75.93	86.85

Analytical samples - 0.70 pounds

SOLVENT RECOVERY. (In pounds.)

Retort	Steaming	Vacuum	Total
1	5.60	0	5.60
2	5.75	0	5.75
3	10.73	0	10.73

Retort 3 was not drained before steaming.0.28 lbs recovered later

ROSIN RECOVERY.

Solvent distilled	201.46 pounds.
Rosin recovered	Not drained.

ANALYSIS OF FRESH WOOD.

Moisture determination.

Retort No.	1	2	3
Sample,gms.	18.8568	18.6400	17.7485
Water,c.c.	6.30	5.85	4.55

Control extraction.

Sample,gms.	17.3100
Dry extracted sample.	10.5220
Rosin by weight	1.2915

SOLVENT ANALYSIS. (Grams rosin per 200 grams solution.)

Solvent	I	II	III	IV	V	VI
In	3.55	2.70	0.85	0.00	0.00	0.00
Out	5.80	6.35	6.10	3.35	2.35	0.50

RUN 11 April 26- May 1 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 45 pounds for 60 minutes.

Vacuum distillation at 20 inches vacuum for 120 minutes.

Extraction at 30 pounds pressure for 15 minutes.

Recovery of solvent.

Steam distillation at 0 pounds for 30 min. and 45 pounds for 60 minutes.

Vacuum distillation at 20 inches vacuum for 120 minutes.

CHARGE. 5/8 inch chips.

Retort No.	Charge
1	37.36 pounds.
2	38.43 " "
3	37.35 " "
Total	113.14

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort	Steaming	Vacuum	Total.
1	135 c.c.	0	135 c.c.
2	90	0	90
3	75	0	75

EXTRACTION. (All data in pounds.)

Solvent	I	II	III	IV	V	VI
In.	79.30	74.57	85.52	76.60	78.38	80.27
Out	64.40	67.16	80.08	81.15	76.64	84.47

Analytical samples 0.75 pounds.

SOLVENT RECOVERY. (In pounds.)

Retort	Steaming	Vacuum	Total
1	2.05	0	2.05
2	2.38	0	2.38
3	6.10	0	6.10

ROSIN RECOVERY.

Solvent distilled	204.79	pounds.
Rosin recovered	12.33	" "

from tests 10 and 11

ANALYSIS OF FRESH WOOD.

Moisture determination.

Retort No.	1	2	3
Sample, gms.	29.8050	27.3850	28.2725
Water, c.c.	8.3	7.1	7.9

Control extraction.

Sample, gms.	17.2455
Dry extracted sample	11.0745
Rosin by weight	1.6860

SOLVENT ANALYSIS.

Solvent	I	II	III	IV	V	VI
In	3.35	2.35	0.50	0.00	0.00	0.00
Out	7.50	7.00	6.20	3.10	2.10	1.05

Note: The moisture content of the sample for the control was 24.23 percent

RUN 12 May 4-8 1915.

CONDITIONS.

Preliminary treatment.

Steam distillation at 45 pounds pressure for 60 minutes.

Vacuum distillation at 20 inches vacuum for 120 minutes.

Extraction at 30 pounds pressure for 45 minutes.

Recovery of solvent.

Steam distillation at 45 pounds for 60 minutes.

Vacuum distillation at 20 inches vacuum for 120 minutes.

CHARGE. 5/8 inch chips.

Retort No.	Charge
1	33.88 pounds.
2	34.14 " "
3	35.65 " "
Total	103.67

TURPENTINE FROM PRELIMINARY TREATMENT.

Retort	Steaming	Vacuum	Total
1	70 c.c.	0	70 c.c.
2	65	0	65
3	65	0	65

EXTRACTION. (All data in pounds.)

Solvent	I	II	III	IV	V	VI
In	80.14	75.80	83.33	82.26	81.52	79.41
Out	63.78	78.85	71.85	77.90	77.27	90.60

Analytical samples 0.75 pounds.

SOLVENT RECOVERY. (In pounds.)

Retort	Steaming	Vacuum	Total
1	2.54	0	2.54
2	3.76	0	3.76
3	5.41	1.10	6.51

ROSIN RECOVERY

Solvent distilled	204.60 pounds
Rosin recovered	not drawn off

ANALYSIS OF FRESH WOOD.

Moisture determination

Retort No	1	2	3
Sample, gms.	28.4830	29.9050	30.0230
Water, c.c.	7.8	8.0	7.7

Control Extraction.

Sample, gms.	18.4200
Dry extracted sample	11.8075
Rosin by weight	1.6910

SOLVENT ANALYSIS. (Grams rosin per 200 grams solution.)

Solvent	I	II	III	IV	V	VI
In	3.10	2.10	1.05	0.00	0.00	0.00
Out	6.40	5.95	5.75	3.70	1.95	0.60

Note: The moisture content of the control sample was 24.96 percent.

Approved:

Robert C. Palmer

Lecturer in Forest Products

Chemist in Forest Products,
U. S. Forest Service

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